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VACUUM

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Saul Dushman 1883—1954

As a tribute from 'Vacuum' to that great pioneering scientist in vacuum physics, Dr. Saul Dushman, whose death on July 7th, 1954 we profoundly regret to record, Dr. Irving Langmuir, a lifelong friend and colleague, and himself one of the outstanding personalities in the field of vacuum techniques, has written the following memorial.

Saul Dushman — A Human Catalyst

A Tribute by Irving Langmuir

THE READERS OF *Vacuum* will suffer a particular loss through the death of Dr. Saul Dushman on July 7, 1954. At the time that he was stricken with his final illness, he was engaged in writing a second volume of his book, *Scientific Foundations of Vacuum Technique*.

To his colleagues in the Research Laboratory of the General Electric Company, Schenectady, where he served 40 years (after 1928 as assistant director) his death comes as a very personal loss, for the dynamic influence of his sterling character had been dedicated to the service of his fellow men. He was regarded as 'the Laboratory's greatest builder'.

Saul Dushman was born in Rostov, Russia on July 12, 1883. When he was nine his family moved to Toronto, Canada. His exceptionally high scholastic record in high school won him the Prince of Wales Scholarship at the University of Toronto, where he graduated in 1904 with a B.A. degree.

He studied physical chemistry at Toronto under Professor Lash Miller, who as an ardent disciple of Wilhelm Ostwald, taught that the Atomic Theory, even if it is sometimes a convenient hypothesis, 'should be employed as sparingly as common usage will permit'.

Just after receiving his Ph.D. degree in 1912 he visited the General Electric Research Laboratory and was most enthusiastic about his first introduction to high vacuum phenomena.

I well remember describing to him shadow casting and rectilinear propagation of atoms evaporated from tungsten filaments in high vacuum. A deposit on the glass which absorbed 20 per cent. of the light, consisted of a layer about 14 atoms deep. Dushman smiled and asked whether I regarded that statement as more than 'a figure of speech'.

Within a few months, while he experimented with electron discharges in high vacuum, it came to him as a marvellous revelation that atomic physics was more than a convenient hypothesis.

He thus developed almost a missionary zeal in helping others to realise the importance of modern physics in any understanding of chemistry and related sciences. Most of his 50 scientific papers and his half dozen books have resulted from his devotion to this ideal. In a similar way he became a human catalyst in

the laboratory bringing together people who would contribute to each other's progress.

He published one important paper in 1915 (*General Electric Review*, 18, 156) which described the development of a high-voltage, high-vacuum diode which could rectify as much as 10 kW of alternating current power at 100,000 volts.

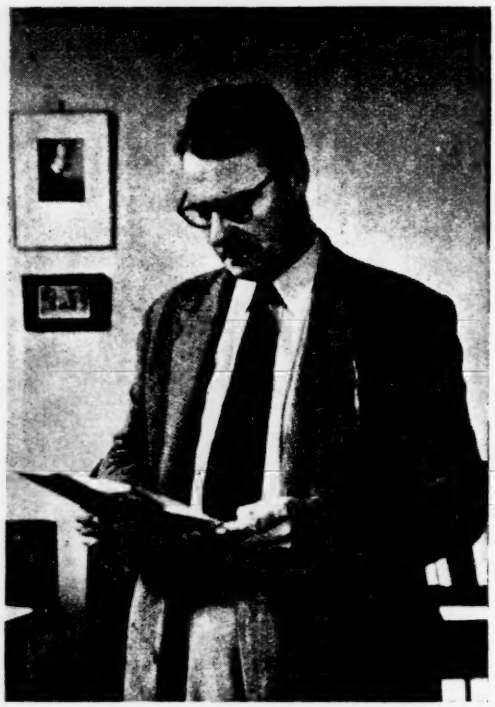
To distinguish devices of this type from former hot-cathode rectifiers, in which positive ions played an essential rôle, a new name was needed. Dr. Dushman and I spent a Sunday morning in 1914 at the home of Dr. Bennett, professor of Greek at Union College, discussing Greek roots that might help us. This marked the birth of the -tron family of vacuum devices. As Dushman said in his paper of 1915, the designation kenotron was ' especially coined from the Greek *kenos* meaning empty, and the suffix *–tron* signifying an instrument or appliance '. The *–tron* family grew into pliotron, radiotron, dynotron, magnetron, thyratron and later into cyclotron, betatron and many others.

A typical example of Dushman catalytic action is the part that he played in the origin of high-speed mercury vapour vacuum pumps. In 1915 Dushman called my attention to the paper that had just been published in Germany describing Gaede's diffusion pump that had a maximum speed of 80 cm^3./sec. He undertook to construct one of Gaede's pumps and studied its behaviour with great care. It was during the discussion of these experiments that the ideas were developed by which the need for the narrow slit in the Gaede pump could be avoided, so that the speed limitation could be removed. Dushman built and tested this condensation pump and obtained in the first model a speed of 3,000 cm^3./sec.

Dr. Whitney, in the early years of the laboratory, had a policy of visiting informally each day with each of the men in the laboratory. Thus a remarkable spirit of co-operation pervaded the laboratory. In later years as the laboratory grew, much close personal contacts became more difficult. Dushman considered it his personal responsibility to perpetuate the 'spirit of the Laboratory'. He recognised the need for bringing together men from different departments. Weekly lunches in the cafeteria, known as ' Dushman luncheons ', were started, at which great care was taken to cut across department lines.

His interest in the problems of his colleagues extended to individuals. He often took the problems of his fellow workers home with him and worked and worried over them as though they were his own.

Schenectady, N.Y.,
October 22, 1954.



FRANK LLEWELLYN JONES

M.A., D.Phil., F.Inst.P.

Professor of Physics,
University of Wales
&
Head of Physics Department,
University College of Swansea

Professor Llewellyn Jones was born in 1907 at Mountain Ash, South Wales, and was educated at West Monmouth School and Merton College, Oxford, where he was an Open Science Exhibitioner. After graduating in Physics in 1929 he was appointed Demonstrator in the Electrical Laboratory under Professor J. S. Townsend, F.R.S., with whom he collaborated in researches in the field of electrical discharges. He also did considerable tuition work. In 1931 he was elected Senior Demy, Magdalen College, Oxford ; and in 1932 was appointed Lecturer in Physics at the University College of Swansea, University of Wales, where he started his investigations on the diffusion of positive ions and on breakdown in gases. Throughout the war he carried out research for the Ministry of Aircraft Production at the Royal Aircraft Establishment where he was in charge of work involving the electrophysics of gases. This related to the spark ignition of aero engines, electrical contact phenomena, and the erosion and behaviour of metals under various discharge conditions ; he was also closely concerned with the development of high frequency ignition systems.

In 1945 he was appointed to the chair of Physics at Swansea, where he has built up a considerable research school in ionisation physics. The researches carried out in his department range over the following fields: Electrical breakdown of gases ; extraction of electrons from electrodes in gases and other secondary emission problems ; discharge sources for quantitative spectrographic analysis ; high frequency discharges; and time studies of ionisation growth. More recently investigations are being undertaken there on the thermal and electrical properties of metals under extreme conditions at temperatures up to their boiling points, such as are found in microscopic molten metal contact bridges, this work being fundamental to the physics of electrical contact phenomena.

Professor Llewellyn Jones has been Dean of the Faculty of Science, and is at present Vice-Principal of the University College of Swansea. He is on the Council of the Physical Society and is a member of the Radio Research Board of the Department of Scientific and Industrial Research. He has also served on the Board of the Institute of Physics, and as Chairman of their Education Committee.

ELECTRICAL DISCHARGES AND THE VACUUM PHYSICIST

F. Llewellyn Jones, M.A., D.Phil., F.Inst.P. *Professor of Physics, University College of Swansea, University of Wales.* *

Summary

THIS PAPER DISCUSSES briefly the various kinds of discharges which can occur in evacuated apparatus, *i.e.*, for ranges of pressure approximately equal or smaller than 10^{-4}mm. Hg when the mean free path is of the order of dimensions of the apparatus. Under these conditions the processes of ionisation in the gas are not always the predominating processes of either initiation or maintenance of such discharges, which are then greatly dependent upon the nature of the electrode surfaces. The mechanisms of the various discharges which can be produced in this way are briefly discussed, and an indication given of the importance of the possible rôles played by magnetic fields and by the nature of the electrode surfaces.

Sommaire

L'AUTEUR CONSIDÈRE les diverses formes de décharges qui peuvent avoir lieu dans une enceinte à vide à des pressions inférieures à 10^{-5} mm. Hg, quand le libre parcours moyen est du même ordre de grandeur que les dimensions de l'appareil. Dans ces conditions l'amorçage et le maintient de telles decharges dépends davantage de la nature de la surface des électrodes que des processus ou des phénomènes d'ionisation dans le gaz. L'auteur indique aussi l'importance du rôle que peuvent avoir les champs magnétiques et la nature de la surface des électrodes.

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INTRODUCTION

AT FIRST SIGHT it might be considered that the subject of electrical discharges through gases is of no great interest to the vacuum physicist and engineer who may be primarily concerned in the elimination of gas from his apparatus. It should be remembered, however, that the term *vacuum* is itself a relative expression which can have different meanings depending upon the particular application. In modern engineering technique the term *vacuum* is often applied to gas pressures in which the mean free path is comparable to the size of the apparatus, *i.e.* pressures $\sim 10^{-4}$ or 10^{-5}mm. Hg, and important applications of such vacuum apparatus involving high voltages have been indicated in this journal previously by Andrade[1]. On the other hand, by the achievements of the modern vacuum physicist, pressures as low as 10^{-10}mm. Hg have been obtained, as shown by the recent work of Alpert[2] and of Biondi[3] in the Westinghouse Laboratories in America, and described previously in this journal in the review by Von Engel[4]. The highest vacua obtainable in a modern laboratory now approach the ideal condition corresponding to a physically negligible quantity of gas molecules.

Gaseous discharges as such have long been of interest to the vacuum physicist because they have been used as an indication of the magnitude of the residual gas pressure, and, indeed, are often used for approximate determinations of the state of the evacuated vessel to-day. For example, at pressures in the mm. Hg range the discharge has the form of a diffuse glow, as distinct from the narrow highly concentrated channel of the spark at higher pressures. For pressures down to 10^{-2}mm. Hg the cathode dark space practically fills the tube and the glass walls fluoresce due to the impact of cathode rays; below pressures of 10^{-3}mm. Hg the conductivity is very low

* MS. received July, 1954.

and a regular discharge only passes with difficulty, but still a discharge can be made to pass.

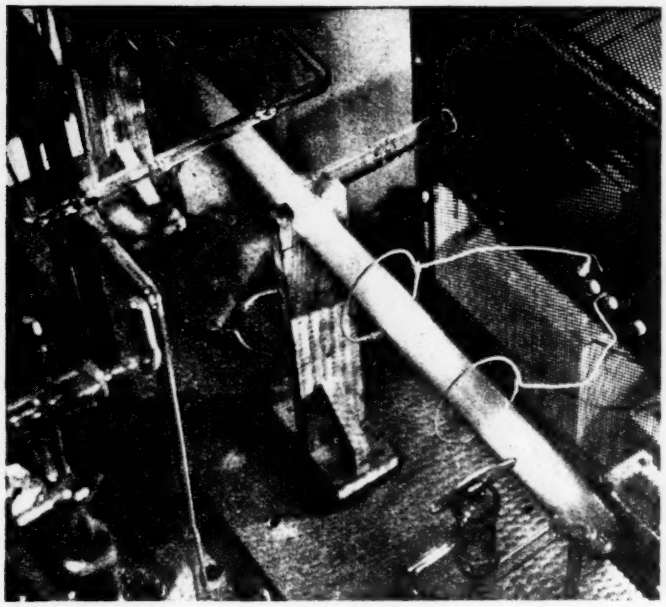
The problem which often faces those concerned with the design and operation of commercial evacuated equipment, such as that of a continuously evacuated cathode ray tube or electron microscope, is that internal flash-over can occur when the required high potentials are applied to the electrodes, and it is interesting to consider how this can happen.

CLASSES OF ELECTRICAL DISCHARGES

It is necessary first to consider the basic principles of the initiation and maintenance of an electrical discharge through a rarefied gas. In order that electrical breakdown can occur two sets of conditions must be satisfied. First, there must be available a sufficient number of initiatory electrons suitably disposed in the inter-electrode space; the optimum position is near the cathode surface. Second, the electrical and geometrical conditions must permit a mechanism of electron current amplification due to ionisation to occur. The rate of ionisation must be such that there is a net increase of electron current after making good the usual loss due to recombination, diffusion and drift in the field. For a gas discharge, the possible mechanisms of amplification are numerous, and they can involve not only the gas but the electrodes themselves. For this amplification of the electron current to occur, the electric field must be sufficiently high in order that the electrons in their transit across the space can ionise a sufficient number of neutral gas molecules by collision (the Townsend primary α process). Further, there must be a process of regeneration by which the electron current can become self-maintained in the absence of a continuous supply of initiatory electrons or after the initial supply has ceased. This regeneration is usually produced by the action of secondary ionisation processes represented separately by the secondary coefficients β, $\alpha\gamma$, δ, ε, η, or simply by a generalised coefficient ω. This generalised secondary coefficient, which is approximately the sum of the separate coefficients, can represent the action of one or more secondary processes acting together[5]. Under these conditions, a discharge, once started by one or more initiatory electrons, can then become self-maintained, or even lead to high current growth even when the initial electrons have been cut off.

The form that the ensuing discharge takes then depends on the nature of the applied electric field. For example, the case of a constant electric field produced by steady potentials applied to various parts of the apparatus is different from the case when an impulse potential is applied to one or more of the electrodes and when the magnitude of the pulse is greater than that corresponding to the minimum potential of static breakdown. In considering whether a discharge will occur between the electrode system under consideration, it is necessary to find whether any of these ionisation processes can take place; the nature of the ionisation processes will, in general, depend upon the conditions in the apparatus.

There are two classes of secondary ionisation processes: Those which occur in the gas, such as ionisation of neutral atoms by high speed positive ions (β process), or the photo-ionisation of molecules by high-energy quanta produced by excitation in the electron current stream (η process); electron emission from the cathode under the impact of either photons (δ process), positive ions (γ process) or excited atoms (ε process), or the emission of positive ions from the anode under the impact of high speed electrons or possibly through local heating. The relative magnitude of these processes will clearly depend upon the conditions in the evacuated vessel. It is clear that, if the gas pressure were low (approximately equal or smaller than 10^{-7}mm. Hg), then ionisation of the residual gas could normally be neglected and it would be necessary to look to electrode processes to effect the necessary secondary ionisation. The emission of electrons from negative electrodes under the impact of radiation, ions or excited atoms is well known and has been studied for many years, and its action in gases has been well appreciated and investigated. Not so much data, however, are available concerning any emission of positive ions from positively charged electrodes, but it is clear that it can be of great importance in producing a discharge when the gas and cathode



An electrodeless high frequency discharge in helium at a pressure of 10^{-4}mm. Hg.

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mechanisms are absent or insignificant. This classification then gives an indication of what class of discharges will be possible in any given set of circumstances. Further, in the case when a high frequency oscillatory field is set up, an initiatory electron can produce an avalanche which is not continually swept to the electrodes as in the case with static electric fields. A discharge can then be produced at low pressures. The photograph above illustrates an electrodeless E discharge[5] produced in helium at a pressure of 10^{-4}mm. Hg. In suitable conditions glass walls can play an important part in the secondary production of the electrons[6]. Finally, it is clear that even when the conditions for the maintenance and development of a discharge are satisfied, no discharge will occur unless suitably disposed initiatory electrons are produced. Since the production of initiatory electrons under various conditions is a most important factor, and as much recent work has been done which has a direct bearing on this aspect, it will be discussed in detail below. One obvious source of initiatory electrons is the action of cosmic rays. The number of electrons produced in this way depends on the volume and inter-electrode space of the vessel under consideration, and for large apparatus it is usually permissible to assume that sufficient electrons are readily available. This conclusion, however, must be modified when sudden impulse voltages are under consideration, because in that case, if the initiatory electrons are to be effective, they must be available at the time when the e.m.f. has reached a sufficiently high value. This problem does not arise, of course, when steady potentials are applied. For small inter-electrode volumes cosmic rays are less effective as a source of initiatory electrons for impulse voltage, but suffice for steady potentials as then sufficient time is available. Before discussing this subject of initiation of the discharge in further detail, it is of interest next to consider the various classes of discharges themselves.

GAS DISCHARGES AT LOW PRESSURES

Consider first the simplest and best known form of electrical breakdown which is that dependent on ionisation in the gas supplemented by electrode effects, *i.e.* electrical breakdown determined by the two classes of ionisation coefficients α and ω under

steady potentials.

The breakdown potential is related to the spark parameter pd, where p is the gas pressure and d is the electrode distance, by the well known Paschen relationship illustrated in Fig. 1. For most diatomic gases the Paschen minimum occurs for a value of pd approximately between 1 and 0.1 cm. mm. Hg. In many industrial applications of evacuated apparatus, the residual gas pressure may be of the order of 10^{-4} mm. Hg or less, and when the electrodes, to which the high potentials are applied, are $\frac{1}{2}$ to 1 metre apart the value of the spark parameter can be of the order of 10^{-2} cm. mm. Hg. Although this is below the value corresponding to the minimum of the Paschen curve, nevertheless, it is not so low that the breakdown potential corresponding to the residual gases normally found in these cases is excessively high.

The breakdown potential can be increased by reducing the spark parameter pd, and, where further reduction of pressure is not practicable, the same result can be obtained by reducing the effective inter-electrode distance d. It would appear therefore that for pressures $\sim 10^{-4}$mm. Hg. an internal flash-over, due to the presence of residual gas, can sometimes be eliminated or rendered more difficult by bringing the electrodes closer together rather than by separating them further provided their leads and back surfaces are suitably insulated. In estimating the probable value of the pressure of the residual gas in any evacuated apparatus from the point of view of the possibility of electrical breakdown, consideration should be given to the evolution of gas inside the evacuated apparatus from the electrodes due to the electron bombardment of the anode and the ion bombardment of the cathode. There is bound to be considerable evolution of gas in this way from un-degassed electrodes.

Another significant factor in discharge initiation and maintenance under these circumstances concerns the value of the electric field and its distribution in the inter-electrode space. This is determined by the geometry of the electrode system and it is clear that sharp curves or points should be avoided on the electrode in order that no considerable concentration of the field should occur anywhere. The electrodes should be shaped so that the field is strongest in the region along the electron path where the field is required (*e.g.* for acceleration purposes), and the electrode and tube walls should be shaped and disposed so that the field intensity falls off, if possible to zero, at first gradually.

The presence of high fields at local points in the electrode surface can be produced by local geometrical irregularities as, for example, by small projecting points or sharp edges, but these can be produced on a microscopic scale in other ways less obvious. For example, an oxidised surface can often be rough; similarly a machined surface, and in fact most cold-worked surfaces, can be microscopically rough unless they have been highly polished. An example of a record of surface finish obtained on a 'Talysurf' machine is given in Fig. 2.* This roughness can lead to a local concentration of the electric field at microscopic points in regions of the surface. This geometrical state, as distinct from the nature of the surface itself, can have an important consequence in initiating and maintaining electrical discharges which, however, has not received sufficient attention. These phenomena will be discussed more fully later, but it is pertinent to point out here that local intensification of the electric field can produce enhanced electron emission from ordinary surfaces effectively making more efficient the ordinary processes of secondary electron emission. In other words the secondary ionisation coefficient γ can appear enhanced. In this way the process assists the development of the pre-breakdown ionisation current which produces the discharge.

Again, quite apart from the geometrical smoothness or otherwise of the electrode surfaces in the vacuum apparatus the nature of the surfaces themselves can play an important part. For example, it is of considerable significance in assessing the electrical activity of an electrode surface to know whether the surface is a clean smooth surface of a pure metal or whether it is the smooth surface of a thin tarnish film. Under these circumstances it is possible for a surface

* See *Measurement of Surface Finish in the Steel Industry*, by M. R. Hopkins (British Iron and Steel Research Association, London).

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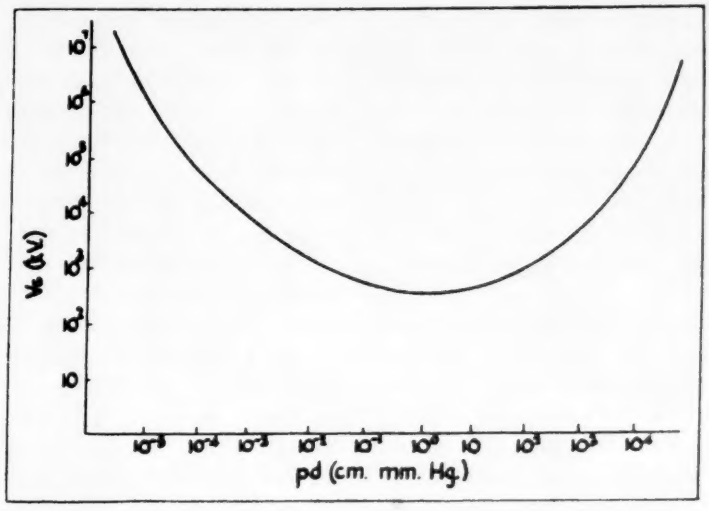
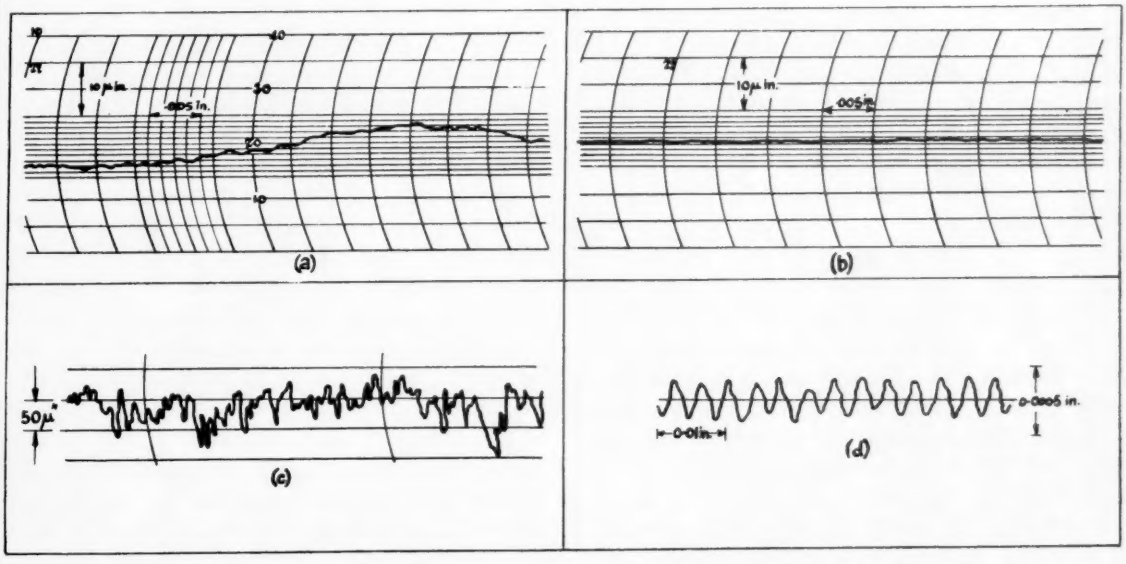


Fig. 1. A typical Paschen static breakdown curve. The region where pd is that corresponding to the minimum V_s is of main interest to the vacuum physicist.



By courtesy of Brit. Iron & Steel Res. Assn.

Fig. 2. Records of typical finishes of steel surfaces obtained on a 'Talysurf' machine : (a) polished stainless steel, (b) plate glass (reference finish), (c) ground surface and (d) turned surfaces.

to show effective electrical activity as manifested by the ability to emit electrons at comparatively low electric fields. This phenomenon again can be of great importance in initiating electrical discharges.

It is of interest next to discuss the influence of magnetic fields on the production and maintenance of electrical discharges under ordinary vacuum conditions when p is $\sim 10^{-4}$ mm. Hg.

INFLUENCE OF MAGNETIC FIELDS

As explained above, for the maintenance of a discharge the value of the spark parameter pd must be sufficiently high. The physical reason for this condition is that there must be a sufficient number of collisions between electrons and molecules of the gas to enable the requisite amount of ionisation to occur in the inter-electrode space. If the pressure is too low then the inter-electrode distance must be correspondingly increased; alternatively, if the electrode distance is small then the pressure must be correspondingly increased. These considerations apply in the absence of a magnetic field; the presence of a magnetic field modifies these conclusions.

This arises from the following simple considerations. Suppose a longitudinal magnetic field H is set up in the space between two electrodes. As a stream of electrons travels between cathode and anode their free paths between collisions are bent in accordance with the relation :

$$Hev = mv^2/r$$

The consequences of this action are that the electron paths in the residual gas are lengthened and also that the lateral diffusion of the electron stream can be reduced, thus reducing electron loss, and the maintenance of the discharge is correspondingly facilitated. This picture applies to the case when the mean free path is smaller than the inter-electrode distance and gas collisions are significant. For lower pressures when the mean free path is of the order of or greater than the inter-electrode distance, the electrons are free in their passage between cathode and anode and the path becomes curved, and the lateral loss can still be reduced.

Another interesting case is that in which a transverse magnetic field is set up parallel to the electrode surface and at right angles to the electric field. This case has been discussed by G. Valle[7] who considered the simple case when all free paths were assumed to be equal. The path of an electron, leaving the cathode normally in its movement down the electric field towards the anode, becomes bent by the transverse magnetic field and is eventually returned to the cathode. In effect, the electron then does not progress towards the anode so that its movement is confined to the space near the cathode as shown in Fig. 3; this phenomenon can be expressed in another way by the idea of *equivalent pressure* which is higher than that obtaining in the vessel. This higher *equivalent pressure* represents the fact that the electrons can now make more collisions with the gas molecules than they would have done in the absence of the magnetic field. The simple theory of Valle was based on the assumption that all electron free paths were equal and this led to certain conclusions which were not in accord with experiment, but a more comprehensive and rigorous investigation was carried out by Somerville[8], who investigated both theoretically and experimentally the consequences of the distribution of free paths about the mean value and electron reflection at the cathode. The fuller theory reached many interesting conclusions which were in accord with experiment. One conclusion reached from the analysis is that for any given actual gas pressure, breakdown cannot be prevented by a transverse magnetic field. The more complete analysis showed quantitatively how the presence of a transverse magnetic field can increase the rate of ionisation and lead to an increased effective gas pressure. The effect of this on the maintenance of a discharge depends, of course, on the actual magnitude of the gas pressure and on the spark parameter pd. For example, if the spark parameter is high, *i.e.* greater than that corresponding to the Paschen minimum, then the transverse magnetic field effectively increases the parameter and makes a discharge more difficult to pass in that a higher potential is necessary. On the other hand, if the spark parameter in the evacuated vessel is small and less than that corresponding to the Paschen minimum,

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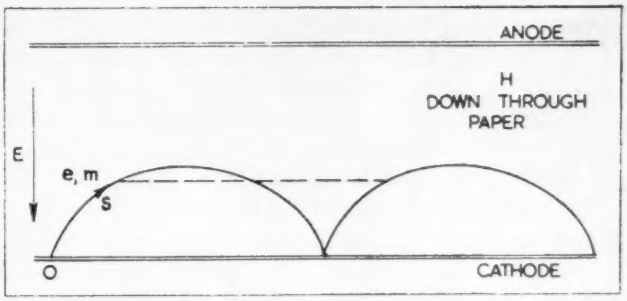


Fig. 3. The effect of a transverse magnetic field is to confine the electrons to the space near the cathode. With reflection from the cathode, the path of the electron will consist of successive arcs of a cycloid.

as is in general the case in vacuum apparatus, then the effect of a transverse magnetic field in increasing the magnitude of the spark parameter would now enable the discharge to pass more easily in that it would require a lower potential. This result is readily seen by reference to Fig. 1.

Thus the above considerations show that the influence of a magnetic field, and indeed the influence of the geometry and value of the spark parameter in any given case, depends on the magnitude of that parameter : The effects are different under different conditions. These remarks are relevant to considerations of internal vacuum breakdown in electronic apparatus such as magnetrons. It is of interest to point out here however that the principles discussed above have been applied in the Philips gauge which utilises this action of the magnetic field and enables measurements of low pressures to be made from reading measurements of the electrical currents produced by the higher effective pressures[4].

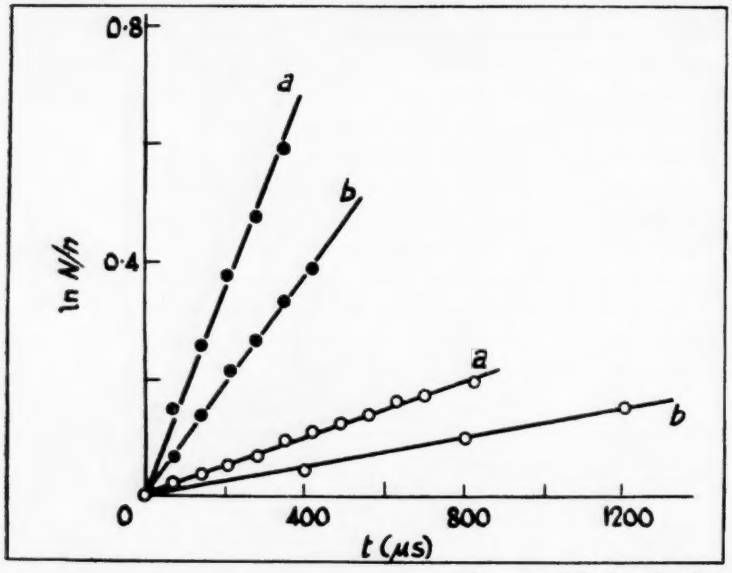
ROLE OF ELECTRODES IN VACUUM BREAKDOWN

It is clear that, if the gas pressure is sufficiently low so that the mean free path is greater than the inter-electrode distance, the gas processes can be regarded as playing no significant part in the ionisation amplification process. Under these circumstances the electrodes themselves are the source of the electrons necessary for initiating any discharge and they also play an important rôle in the mechanism of the build-up and maintenance of a discharge.

It has long been known that vacuum breakdown, often leading to destructive arcs, can occur under conditions when it is not easy to see where the initiatory electrons come from or to understand the mechanism of maintenance. This is well known in particle accelerators, magnetrons, vacuum contact devices, vacuum condensers, X-ray tubes for deep-ray therapy and many other applications; it is not improbable that the wellknown phenomena of back-fires in mercury arc rectifiers may also be related to the same basic problem. These electrical discharges occur when the electrodes themselves are cold and the macroscopic electric field near their surfaces is no greater than about 10^5 V cm. and the specific physical problem which arises is due to the fact that these fields are orders of magnitude too low to account for the emission on the basis, for example, of the Fowler-Nordheim theory of electron emission from metals. This problem of the cold emission of electrons under comparatively low electric fields has been the subject of much investigation in recent years.

For many years it has been appreciated that the nature of the electrode itself plays an important part in determining its electrical activity and it is pertinent here to note that in most industrial applications of so-called vacuum electrical apparatus where internal breakdown occurs the electrode surfaces are not those of perfectly smooth pure metals. Inevitably they are covered with at least thin ($\gtrsim 10^{-7}$cm.) tarnish films, while in some cases thick ($\gtrsim 10^{-5}$cm.) oxide layers are present. Further the electrode surfaces are themselves often far from microscopically smooth, this being especially the case when the electrodes are not polished to any considerable degree after machining. There is a large body of evidence to indicate that the electrodes in such a state have certain electrical activity in gases in the presence of an electric field. See for example, the work of Paetow[9] in Germany, Slepian and Berkey[10] in the U.S.A.; and in work

carried out during World War II in this country such effects[11] were applied to produce electrically active surfaces when required, for instance, in self-triggering spark gaps. In the realm of low-voltage phenomena, work[12] on electrical contacts at the Bell Telephone Laboratory in the U.S.A. has indicated that the nature of the electrode surface has a highly significant influence on the production of electrons which can initiate and maintain an arc when two electrodes approach even at low voltages < 100 V. In all these cases, however, there appeared still to be an outstanding problem. The electric field at the surfaces from which electrons appeared were still orders of magnitude lower than that required by the Fowler-Nordheim theory of cold emission from metals.



By courtesy of The Proc. Roy. Soc.

Fig. 4. Graphs of lnN/n against t. (a) nickel cathode; (b) tungsten cathode; ●—oxidized; o—polished. $E = 10^5$ V/cm.

A discussion of cold electrode emission is therefore of interest in connection with the possible rôle of electrodes in vacuum breakdown.

ELECTRON EMISSION FROM COLD ELECTRODES

Experimental investigations of this problem which have been made in recent years in the Physics Department, University College of Swansea,[13, 14, 15] have been concerned with the functional dependence on the electric field of the electron emission from various cold electrode surfaces in gases. The method used, involved the determination of the mean statistical time lag of spark breakdown, which analysis shows to bear a simple relation to the rate of pre-breakdown electron emission from the cathode. In this way the cold electron emission from various electrode materials in different surface conditions was investigated for a range of applied macroscopic electric fields in various gas atmospheres. The technique depended upon the application of a succession of unit function pulses of electric force and the liberation of a single electron was detected electronically from the collapse of the applied voltage and the time lag recorded; from this the mean was easily found. A simple relation enables the rate of electron emission under the given field to be obtained from the slope of a linear graph of the logarithm of the distribution N/n of measured times t. Typical graphs are given in Fig. 4. By obtaining such graphs for different values of the macroscopic applied field E_m the functional dependence of I upon E_m may be found for different electrodes. The relationship so found is illustrated in Fig. 5 which shows it to be of the same form as that of the Fowler-Nordheim equation for field emission. Interpretation of the graph on this basis enables estimates of the *effective* work function of the apparent source of the electrons to be made. The method provided a convenient means of readily testing different surfaces.

Metals in a variety of surface states were examined. These included nickel, tungsten, iron, copper and aluminium and alloys in different surface conditions corresponding to :—Normally oxidised surfaces due

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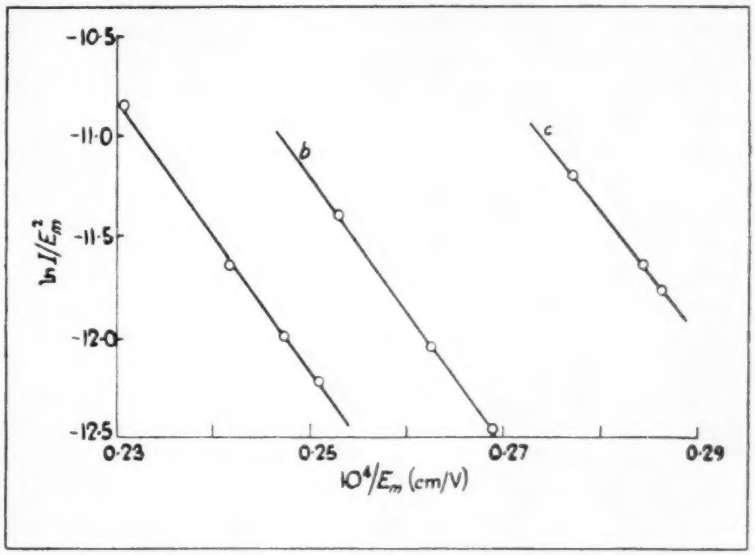
to long contact with air; different surface finishes, such as rolled, ground or polished to a very high degree; and finally surfaces which have been cleaned by bombardment in a hydrogen glow discharge and outgassed in a vacuum. The experimental results showed that these different surfaces had very different degrees of electrical activity and it seemed possible to classify the mechanisms of emission into three kinds. Firstly, substantial emission from ordinary surfaces occurred with moderate electric fields $\sim 10^5$ V/cm. with comparatively thick ($\gtrsim 10^{-5}$cm.) and rough oxide layers. Secondly, the emission from surfaces covered with tarnish layers in dry gases appeared to be due to a different mechanism. Finally, the emission from smooth clean surfaces appeared to be negligible with these low fields which were able to produce high emission from the other surfaces; and it would appear that for smooth perfectly clean surfaces very much higher ($>10^2$) fields are required to produce significant emission. Results obtained in these experiments with metals covered with oxide layers if interpreted in the Fowler-Nordheim equation were consistent with the view that the electrons were extracted from sites situated on the oxide surface which had areas $\sim 10^{-14}$ cm.^{2} and *effective* work functions $\sim$ 0.5 eV. With electrodes which had been exposed to air or oxygen, it is likely on this interpretation that in those cases the emitting sites were of molecular dimension, and might in fact, be groups of negative ions. Seeger[16], in Germany, studying electron emission from surfaces covered with aluminium oxide layers as an after-effect of irradiation and bombardment, also obtained enhanced emission and deduced estimates of the work function of the source of this emission. His estimates were of the order of 0.62 eV, and he concluded that single atoms gave rise to this emission.

The great effectiveness of the process of the cold emission of electrons as a mechanism for the initiation of discharges, lies in the fact that the electrons are produced in the optimum position (at the cathode) and at the correct time (when the high field is present).

ROLE OF TARNISH FILMS

The same time lag technique showed that the above theory of extraction of electrons from surfaces with oxide layers did not apply to tarnished surfaces in dry gases. Briefly no physically significant values of emitting area and work function of the emitting sites could be obtained by application of the Fowler-Nordheim theory. Further investigation indicated the possibility of an important rôle being played by positive ions and the tarnish film. For example, using a dry gas an electric field considerably greater than the static sparking field could be applied to the discharge gap for times up to minutes without the initiation of a discharge. However, as soon as one discharge had occurred subsequent discharges could always be



By courtesy of The Proc. Roy. Soc.

Fig. 5. Dependence of I on E_m for various surfaces. (a) nickel; (b) steel; (c) aluminium-magnesium alloy.

obtained immediately on re-application of an impulse of potential which was only slightly greater than the static sparking potential. This indicated the influence of the first spark upon the initiation of subsequent sparks and it appeared likely that ionisation remaining in the gap was responsible for the initiation of the next spark. If positive ions produced in the first discharge could be partly retained on an insulating film on the cathode, an intense electric field could be set up across this film at the metal surface. The potential barrier presented to electrons within the metal would then be distorted and lowered, and the escape of electrons assisted. The dependence of the observed electron emission on the applied field has been discussed[14,15] in terms of a modified Fowler-Nordheim theory. Further work on the interaction of positive ions on such insulating surface films is needed to clarify the mechanism and such an investigation is now in progress. The action of both positive ions and tarnish films seems to be necessary for this mechanism of electron production. When the film was practically removed (by hydrogen ion bombardment in a glow discharge), the electron emission obtained under electric fields $\sim 10^4$ to 10^5 V/cm. from clean surfaces was negligible. For perfectly clean surfaces the emission requires fields of magnitude predicted by the Fowler-Nordheim theory, as shown by the recent comprehensive investigations of field emission from clean metal electrodes *in vacuo* by Dyke and Trolan[17], Dyke, Dolan and Trolan[18]; Dolan and Barnes[19]; Dyke, Trolan, Martin and Barbour[20], who have carried out a series of investigations using clean outgassed metals under high vacuum conditions.

Increasing interest has in recent years been shown in the fundamental processes, which lead to arcing at electrical contacts[21]. At the *break*, or separation of contact members, the question of the source of initiatory electrons offers no difficulty owing to the intense thermionic emission caused by the molten metal bridge formed between the separating contacts. At *make*, however, the source of electrons which could initiate a discharge between the cold contact members on the point of closing is less obvious. Under such conditions the nature of the cathode surface can be of very great importance. The emission phenomena outlined above and the mechanism of electron production can lead to the increased probability of arcs forming at opening contacts. In the case of the oxide film[21] enhanced emission can lead to arcing; and Germer and his colleagues[12,27] have demonstrated the influence of positive ions on contaminated electrodes in initiating arcs in gases. It is possible that the process of electron production discussed above also occurs at very low gas pressure; if so, it would be an important contributory factor in initiating vacuum breakdown. Very recent work by Kerner and Raether[28] has demonstrated emission from cold electrodes at fields $\sim 10^4$ V/cm. in air at $\sim 10^{-5}$mm. Hg.

SOME EXAMPLES OF VACUUM BREAKDOWN

For the insulation of the high voltage electrodes using gas pressures in which the mean free path is of the order of the electrode dimensions, difficulties have been encountered owing to the irregular and sometimes spasmodic occurrence of internal flash-over and the mechanism by which this discharge occurs has not yet been completely elucidated. In many cases such internal failure and breakdown occurs, even when the pressures are so low that the 'gas discharge' breakdown corresponding to that value of pd, requires potentials far in excess of those applied. Clearly then the mechanism must be very different from those considered in the previous sections, and the breakdown is not a gas discharge phenomenon in that sense. Some other mechanism of electron production and current amplification must therefore be sought. With a large apparatus there is no great difficulty in finding a source of initiatory electrons. The breakdowns which are observed are irregular and spasmodic and occur at intervals which are satisfactorily accounted for by the appearance of electrons produced by the incidence of cosmic rays on the apparatus and by other natural sources. The main difficulty is to account for the development of ionisation rather than for its initiation. In early investigations of this kind of

phenomena pointed electrodes were often used and the nature of the residual gas was often unknown and was at a substantial pressure; these conditions together with the presence of contamination in the electrode surfaces made it very difficult to reach any quantitative results. Very recent work, however, carried out by Calvert[22] who used uniform fields with electrodes which had been exposed to the air, appears to indicate that cold electron emission is first produced from the cathode by the field and this result is in accordance with work described above. In addition, however, further amplification is noticed and this may occur in a vessel where the mean free path is of the dimensions of the inter-electrode space. The effects of the impact of electrons accelerated through the anode potential on impact with the anode cannot be neglected and the results so far appear to indicate that there is a possibility of positive ions being emitted from the anode under these conditions.[23, 24, 25]. Such a mechanism if it occurs would certainly account for rapid amplification because the high speed ions would have no difficulty in liberating electrons on impact which would then build up the process. Under fully controlled conditions when the micro-geometry is known and the metals are outgassed and pure the recent experiments of Dyke *et al.*[20] have demonstrated that the production of electrons is a cold field emission process in full accordance with the Fowler-Nordheim theory and no evidence was found involving any matter transfer from the anode. Under normal industrial conditions, however, involving oxidised and non-degassed surfaces the question of matter transfer from the anode, which would involve an apparent dependence of current on total voltage as distinct from the field at the cathode might therefore be involved. Clearly further work is required in this interesting field.

Another important aspect of this incipient breakdown is that of the electrical breakdown across the surface of insulators and has been the subject of investigations by Gleichauf (1951)[26].

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THE ELECTRONIC CLEAN-UP OF GASES

in sealed-off vacuum systems

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Summary

SOME OF THE previous work in this field is reviewed briefly, and a few of the conflicting theories put forward to account for the observations are mentioned. The results of fresh experiments, using sealed-off tungsten filament triode ionisation gauges, are described. In the cleaning-up of gas in these tubes, two regions of the log P *vs.* time curves are distinguished. The speed of pumping or cleaning-up of gas in each region is constant, and hence the rate of cleaning-up of gas is proportional to pressure. In the first or ' A ' region of the falling pressure/time curves, the speed of pumping is a few cm.^{3} sec^{-1}; in the second or ' B ' region, the speed is about 1/20th as great. In the ' A ' region, the speed of pumping is proportional to the ionising electron current, but in the ' B ' region it is independent of electron current for values greater than a few tens of microamperes. It has been found that the cleaned-up gas is held by the glass walls of the tubes, particularly in the vicinity of the electrode structure. When all the glass was shielded from the products of the ionising electron current, clean-up of gas ceased. The significance of the two regions of the clean-up curves is discussed, and possible mechanisms are suggested in keeping with the observations in the greater part of the present experiments, and those reported by previous workers.

Sommaire

ON FAIT UNE brève récapitulation des travaux dans ce domaine et on mentionne quelques-unes des théories proposées pour expliquer les observations. On décrit ensuite les résultats de nouvelles expériences faites au moyen d'une jauge à ionisation scellée, à filament de tungstène. On peut distinguer deux parties dans la courbe log P en fonction du temps pour le phenomène d'abaissement de la pression durant la décharge. Dans la première partie de la région ' A ' de la courbe, la vitesse de pompage est de quelques c.c./sec. tandis que dans la partie ' B ', elle est d'à peu près un vingtième de cette valeur. En plus la vitesse de pompage est proportionelle au courant électronique ionisant dans la partie ' A ', mais est indépendante de ce courant dans la partie ' B ', pour des valeurs supérieures à quelques dizaines de micro-amperes. On a trouvé que les gaz sont retenues par les parois de verres du tube particulièrement à l'entour des électrodes. En ' protegeant ' le verre des produits du courant ionisant, on fait cesser l'abaissement de la pression. L'auteur discute la signification des deux regions de la courbe et suggère quelques hypotheses du phenomène, que pourraient expliquer les observations de ces récents travaux, ainsi que de travaux antérieurs effectués par d'autres chercheurs.

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INTRODUCTION

IT IS WELL KNOWN that when an electronic discharge takes place in a sealed vacuum vessel, a continual clean-up of the residual gases results. In particular, in a hard valve, the presence of an electron beam with energy greater than about the ionisation potential causes gas in the valve to clean-up with a corresponding reduction in pressure. This effect is of the greatest importance in radio valve engineering although, because the effect normally works to the advantage of the manufacturer, a full understanding of its mechanism may not be considered of paramount importance. Nevertheless, present knowledge of the detailed mechanisms involved in processing valves is very scanty, and a fuller understanding of these becomes more important as more stringent require-

* MS. received May, 1954.

ments are laid down for the valves.

The use of very high vacua (pressure $<10^{-10}$mm. Hg) is at present mainly of importance in research investigations, although it will inevitably be of increasing practical importance as time goes on. Such low pressures are required because it is often desired to measure a property of a surface in the absence of even a monolayer of gas, and at relatively low temperatures. It is often possible to clean a surface of an adsorbed monolayer by heating or by bombardment but the classical kinetic theory of gases predicts that a new layer forms again on cooling in a time given by :

$$t = \frac{2.4 \times 10^{-6}}{P} \quad \text{(sec.)}$$

where P is the gas pressure in mm. of. mercury, assuming that the gas is oxygen, that the condensation coefficient is unity, and that the molecules in the monolayer are packed as closely as possible. To make this time long enough to carry out an experiment, before appreciable contamination has condensed, thus requires a pressure of about 10^{-10} mm. Hg.

A good review of previous work on clean-up is given by Dushman[1]. The results of the reported work are conflicting. It is well established that clean-up only occurs when an electron beam is present and with an energy greater than that to cause ionisation of the gas present. Strong evidence has been put forward that the gas is cleaned-up on (or in) the glass walls of the tube, although this has been by no means certain, see von Meyeren[2]. It has been suggested that the mechanism of the clean-up process does not involve the ionisation of the gas, but rather the dissociation of the molecules to form active nascent atoms, see Hughes[3]. The clean-up of residual gases in gettered radio valves, with oxide-coated cathodes has been described by Metson[4]. Triodes with tungsten filaments were also used in Metson's experiments. It was assumed by this author that ions were cleaned-up to the ion collector and the cathode. This paper describes some experiments and observations which, though not fully elucidating the clean-up mechanism, bring out new facts which may subsequently help to provide an adequate explanation.

EXPERIMENTAL ARRANGEMENT AND PROCEDURE

To illustrate the clean-up phenomenon, a simple experiment, which has been repeated many times, is described.

A tungsten filament triode ionisation gauge was sealed off after being baked and outgassed. The pressure after sealing-off was 10^{-5} mm. Hg. The gauge was then run with 1.25 milliamperes of electron current, the ion current being read periodically, and the equivalent air pressure deduced from these readings. Normally, the ion collector potential was between zero and + 1 volt, the filament potential was + 30 volt and the electron collector potential + 210 volt. If the clean-up process is a simple one, it will be expected that the rate of clean-up will be proportional to the concentration of molecules present, that is to the gauge reading. Thus, the pressure would follow the law :

$$P = P_o e^{-ct}$$

where P_o is the starting pressure, c a constant and t the time from starting. If the readings are plotted as log P against time, a straight line of negative slope would then result. Fig. 1 shows a typical plot of this sort. It is seen that two regions occur. In the first, or 'A' region, the slope of the curve is approximately twenty times that in the second, or 'B' region. After some time, the slope of the 'B' region lessens until a final equilibrium pressure is reached. The presence of these two regions of the clean-up curve has not been reported before.

No significant change in the shape of the clean-up curve resulted from changes in processing before sealing-off. Tubes baked by radio frequency or conduction heating, and pumped with mercury or oil condensation pumps, with or without liquid air traps, all gave similar characteristics. The sealing-off pressure also had no marked effect on the subsequent shape of the clean-up characteristic; the fraction of the initial pressure at which the knee between the regions 'A' and 'B' occurred, being always the same ($\pm 20\%$).

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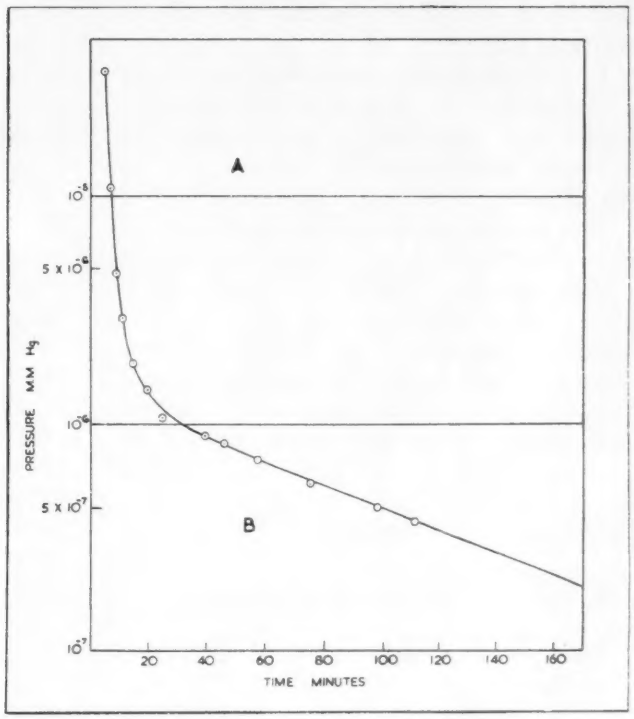

Fig. 1. A typical log P *vs.* t clean-up curve, showing the two linear regions 'A' and 'B'.

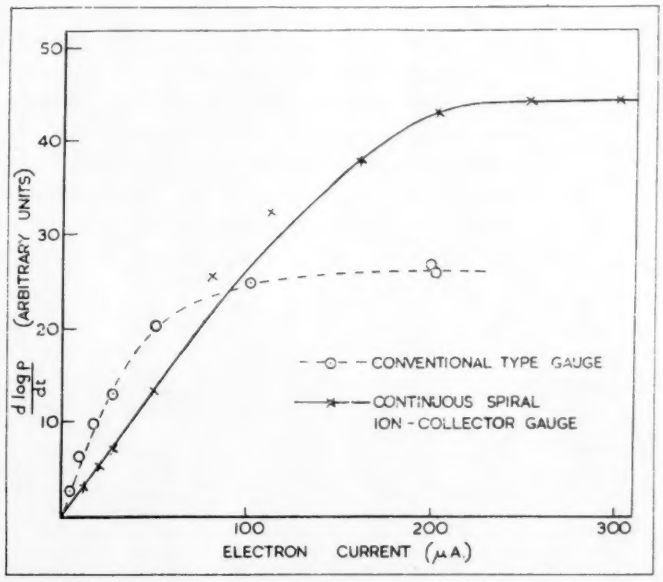

Fig. 2. The variation of speed of pumping with electron current for region 'B' of clean-up curves.

Experiments were made on tubes of three different geometries, *viz.*:

(i) A conventional cylindrical sheet anode triode with the plate used as an ion collector.

(ii) A gauge with sheet anode replaced by a continuously wound wire spiral.

(iii) A gauge designed to reduce the number of photo-electrons emitted by the positive ion collector under soft X-ray bombardment (Bayard and Alpert[5]) and capable of reading pressures down to 10^{-10}mm. Hg.

Clean-up characteristics were measured for these tubes and found to be practically identical. That is to say, the rate of fall of the logarithm of the pressure and the fraction of the initial pressure at which the A-B knee occurred was constant. The pressure in each case was measured by the tube itself after previous calibration against a standard tube.

EVALUATION OF OBSERVATIONS

Dependence of Clean-up Rate on Ionising Current

If the clean-up process is dependent on the formation of ions, it is to be expected that the rate of clean-up will vary directly with the ionising electron current. Experiments to confirm this showed that in region A, a linear dependence occurred, whereas in region B the rate of clean-up was only proportional to electron current for currents below about 50 microamperes (see Fig. 2). Above this, up to the normal operating current of 1.25 milliamperes, and on up to 10 milliamperes, the rate of clean-up was independent of electron current. Such a result has not been reported by previous workers. This observation led to a comparison between the number of molecules cleaned-up, as measured by the pressure drop, and the number of ions formed, as measured by the ion current collected. The ratio between these is termed the clean-up efficiency (η_c). This efficiency is given by :

$$\eta_c = \frac{1.59\ V\ K_g}{10^9\ k\ T} \times S$$

where V is the volume of the gauge (c.c.), K_g the gauge sensitivity factor (μA per mA per micron Hg), S the slope of the log P *vs.*t. curve (logs to base 10, time in minutes), k is Boltzmann's constant, and T the absolute temperature. For the conventional gauge, of the sheet ion-collector type, used at a temperature of 300°K,

$$\eta_c = \frac{40 \times S}{I_e}$$

where I_e is the electron current causing ionisation (in mA).

There are two sources of uncertainty in this relation. The measured ion current does not necessarily include all the ions produced. Some of these are lost outside the collector, and some are produced between the cathode and the electron accelerator electrode and are accelerated to the cathode rather than to the ion collector. The ratio of the ions collected to the total ions produced between the accelerator electrode and the collector was measured for the types of gauge used, and hence a correction made to include this total number in the value of K_g used in the efficiency formula. The new value of K_g is denoted by K'_g, and the new efficiency is called the overall efficiency. The ions accelerated to the cathode are still not included. The second uncertainty arises because the quantity of gas cleaned-up is calculated from the fall in pressure on the assumption that no gas is liberated from the tube walls and electrode surfaces as the pressure falls. This assumption is unwarranted in region A at least, as will appear later. The efficiencies measured for a series of tubes and for regions A and B are shown in Table I.

Table I

(Electron Current = 1.25 mA).

Gauge Type	*Region of Clean-Up Curve*	*Fractional Efficiency*	*Overall Efficiency*
Conventional Sheet Ion-Collector	A	0.034	0.034
	B	0.0033	0.0033
Open Spiral Ion-Collector	A	0.043	0.0086
	B	0.0037	0.00074
Anti-X-Ray	A	0.41	0.065
	B	0.0215	0.0034

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It will be seen that the clean-up efficiency is apparently quite low. It is important to consider whether this is inherent in the clean-up process or is due to the fact that gas is desorbed from surfaces as the pressure falls. If the latter is the case, then it results from the gas in the vapour phase being in equilibrium with the gas adsorbed on the exposed surfaces. If this is so, a strong temperature dependence of the pressure will be expected, since the rate of desorption will increase roughly exponentially with temperature, whereas the rate of adsorption or surface bombardment is not greatly affected by temperature if the pressure remains constant. Such a temperature dependence was found in region A. In fact, this dependence was so marked that precautions had to be taken to obtain curves showing a logarithmic dependence of pressure upon time of running. In region B, the measured pressure was very little affected by the tube temperature. Thus, the inefficiency in region A can be explained, at least in part, by the desorption of gas from the walls.

In region B, as has been stated, the clean-up is independent of electron current above about 50 microamperes; therefore the efficiency will be greater at low electron currents. For electron currents less than about 50 microamperes, the efficiency has a constant value, approximately 10% in the conventional cylindrical sheet anode type of gauge.

Location of Cleaned-up Gas

It is of especial interest to know the location of the cleaned-up gas. It has previously been suggested that this goes to the glass (Campbell[6] and Schwarz[7,8]) During the course of experiments, it had been found that after clean-up in the B region, heating of the glass at the end of the bulbs liberated very little gas, even up to temperatures of 100-200°C. However, heating the glass near the ion gauge structure liberated gas after quite a small temperature rise. This suggested the clean-up sites to be in this region of the glass. It was possible to find the extent of the clean-up sites by exploring the glass surface with a fine flame or hot wire. In this way, it was shown that the gas was attached to the glass surface over an area near the ends of the nickel cylinder in the conventional gauge, and over a region extending over the length of the collector in the spiral collector and anti-X-ray tube. A special tube was constructed with an internal 'Aquadag' coating over the inside wall (see Fig. 3), and no measurable clean-up occurred. This tube was broken open, and a narrow gap 2 cm. wide made in the 'Aquadag' coating in the region X-Y of Fig. 3. Clean-up occurred at the full B rate. It is a remarkable fact that, though the clean-up appears to be on the glass walls, and though it can

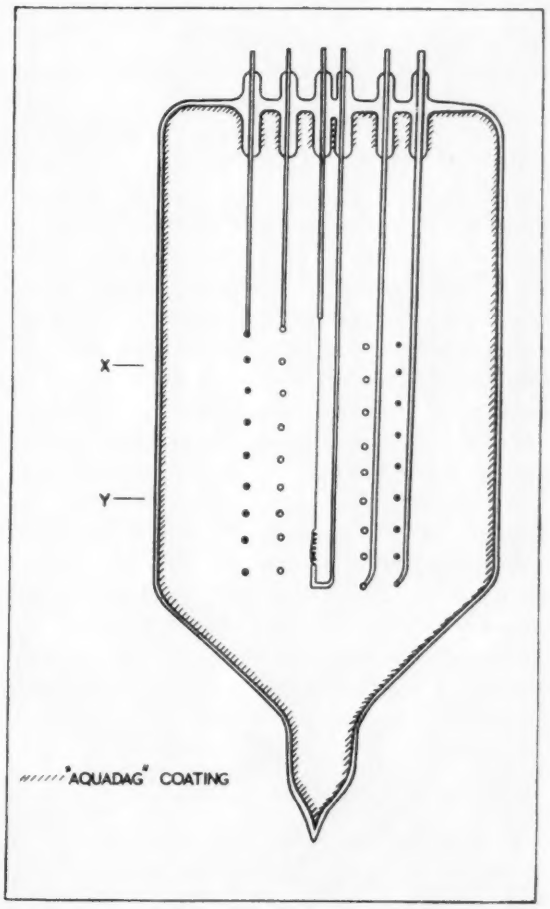

Fig. 3. Schematic diagram of experimental tube with 'Aquadag' coating.

be stopped by shielding the walls of the gauge, either by an ' Aquadag ' coating on the glass or by enclosing the ion gauge electrodes within a metal (molybdenum or nickel) can, the partial shield formed by the sheet collector in the conventional gauge has no effect on the clean-up rate.

Significance of the Regions A and B in the Clean-up Curve

It might be assumed that the two regions correspond with the clean-up of two different gases, since previous workers (for example, Campbell[6] and von Meyeren[2]) have found that different gases clean-up at different rates. However, it was thought possible that the two regions correspond to two different clean-up mechanisms, the first mechanism saturating, for some reason, after the adsorption of a certain quantity of gas. This immediately seems unlikely, since the fractional drop in pressure over the A region is not dependent on the starting pressure, hence the knee of the curve corresponds to different quantities of gas cleaned up in the various runs. Furthermore, if after a clean-up run further gas was evolved, from an electrode for instance, a subsequent clean-up run had identical characteristics to the initial one. If, on the other hand, a tube was allowed to stand with the cathode cold for a time after having been cleaned up, it was found that a small pressure rise resulted. The subsequent clean-up curve had a much smaller ratio of initial pressure/knee pressure. This suggests that gas is re-evolved from the sites to which it is cleaned-up, and that the B gas is less tightly bound than the A gas, so that more B gas is evolved. Alternatively, the rise in pressure could be caused by helium diffusion through the glass walls (Barrer[9], Alpert and Buritz[10]). If this is so, the B gas is probably helium.

DISCUSSION OF THE CLEAN-UP MECHANISM

The basic experimental results, whose mutual incompatibility upsets any simple theory of the clean-up mechanism, are chiefly these :—

(*a*) The rate of clean-up is independent of the electron currents above a few tens of micro-amperes.

(*b*) The rate of clean-up is proportional to pressure.

It has, of course, been checked that the rate of ion formation is proportional to the electron current and the concentration of gas molecules in the bulb. If it is assumed that the spatial distribution of electron and ion density is not affected by the value of the electron current, then the mechanism apparently must include some connection between the final clean-up process and the un-ionised gas molecules other than the obvious one *via* the ionising process. That is to say, the clean-up process ' is aware of ' the gas pressure but not by means of the rate of ionisation, for it is insensitive to variations in this. The fact that the rate of clean-up is dependent on the gas pressure rules out the hypothesis that there are a limited number of sites into which the ' ions ' can be cleaned up, and that these are renewed by, for instance, diffusion at a limited rate. Such a hypothesis would explain the independence of clean-up of current, but not the linear dependence on gas pressure unless it be assumed that the number of sites is affected by gas pressure. This seems unlikely, but is not altogether impossible.

Schwarz[7] has suggested that the state of charge of the wall is of prime importance and, indeed, his experiments seem to indicate this to be true for the clean-up of gases in the type of tube on which his experiments were carried out. Some experiments were carried out in an attempt to ascertain the effect of charge. Large potential differences were applied between the outside of the glass and suitably placed electrodes inside. No effect on clean-up rate was observed. These results suggest that the clean-up is indeed due to activated molecules (dissociated or metastable). It would be expected that the rate of formation of these activated molecules would still be proportional to electron current, so that all the above arguments are still valid.

A possible alternative mechanism could conceivably be involved. The active molecules could be produced not in the gas phase but at the surface of the electron collector, the gas molecules here being activated and desorbed under the action of the

bombarding electrons. Under these conditions, if it is assumed that every adsorbed molecule is quickly struck by an impinging electron at normal electron beam currents, then the rate of production of active molecules will be independent of electron current. At very small electron currents, however, there will be a deficiency of bombarding electrons, and the rate of production of active molecules will tend to be proportional to the electron current. The number of adsorbed molecules will be proportional to pressure. This hypothesis thus explains the saturation of the rate of clean-up with electron current, while not contradicting the observed linear dependence of the rate of clean-up with pressure. The observed independence of the rate of clean-up of exposed glass surface area, which seems to hold down to very small areas, after which no clean-up occurs, can be explained on the basis that the active gas molecules 'bounce' around until they find an exposed glass surface on which to be adsorbed. In a study of getters, Wagener[11] has found such behaviour with nitrogen.

An objection to the theory that the active gas molecules are desorbed from the electron collector is that the rate of clean-up is independent of gauge temperature (in region B at least). The number of adsorbed molecules will be expected to vary exponentially with temperature. Experiments were carried out in which the electron collector temperature was varied by a conduction current. This resulted in no appreciable change in clean-up rate. This independence of temperature might be explained if a complete monolayer of gas were present on the collector, as might indeed be expected with, for example, nitrogen on molybdenum at normal temperatures. However, under these conditions, linear dependence of clean-up rate on pressure would not be expected.

It must therefore be concluded that this at first promising theory is not yet acceptable, though it appears to agree with the experimental results as well as any other theory so far put forward.

The important part played by the activated molecule is confirmed by the observations of Lukirsky and Ptizyn[12], Reimann[13,14] and later Wagener[11] in experiments on clean-up with getters in the presence of an electron beam. It is significant also that Wagener's[11] results show a non-linear dependence of clean-up rate on electron current.

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PREPARATION OF SPECIMENS

for the electron diffraction camera

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Summary

THE PREPARATION of every material for the electron-diffraction camera is a special case, the methods employed depending upon (1) the material and (2) the object of the investigation. It is not possible therefore to give a comprehensive paper covering all possibilities and the method of the article is to illustrate by examples the types of technique used and the precautions which must be observed. These, together with the literature references, are designed to enable the reader to devise suitable methods for any specific case. The subjects are dealt with under the following headings :—*Transmission:* Making Collodion Films ; Powders, Suspensions and Deposits from Solution; Rubber, Oils, Greases ; Metal Films ; Oxide Films and Vitreous Substances. *Reflection :* Powders, Smokes, Crystals, Condensed Vapours ; Oils, Greases ; Compact Solids, Especially Single Crystals; Polycrystalline Solids. There are also sections on reactions carried out in the camera and on precautions.

Sommaire

LA PRÉPARATION DE CHAQUE spécimen pour le diffractographe électronique présente un cas spécial, les méthodes à employer dépendant (1) de la substance, (2) du but de la recherche. Il est donc impossible de donner un article comprenant tous les différents cas. La méthode de l'auteur est d'illustrer par des exemples les différentes techniques et les précautions à observer. Celles-ci ajoutées aux références bibliographiques ont pour but de permettre aux lecteur de choisir les méthodes apropriées à chaque cas particulier. Les questions sont traitées dans les paragraphes suivants; *Transmission:* Préparation des Films de Collodion; Les Poudres; Les Suspensions et Dépots à Partir de Solutions; Les Caoutchoucs; Les Huiles; Les Graisses; Les Couches Metalliques; Les Couches d'Oxyde; Les Verres. *Reflexion:* Les Poudres; Les Cristaux; Les Vapeurs condensées; Les Huiles; Les Graisses; Les Corps Denses; en particulier les Monocristaux; Les Solides Polycristallins. Il y a aussi des paragraphes sur les réactions qui peuvent se produire dans le diffractographe et les précautions à prendre.

INTRODUCTION

THIS ARTICLE IS NOT INTENDED as a review but rather as an introduction to supplement the literature on the subject. The approach used is to select a series of typical examples in the belief that the reader will then, with the help of the appropriate literature, be able to devise methods for particular cases. It is therefore a collection of hints, suggestions and cautions, some of which are difficult to embody in scientific papers but which, nevertheless, are useful and important.

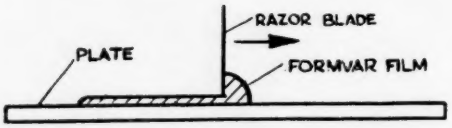
* MS. received June, 1954.

There are two methods of examining specimens in the diffraction camera :—

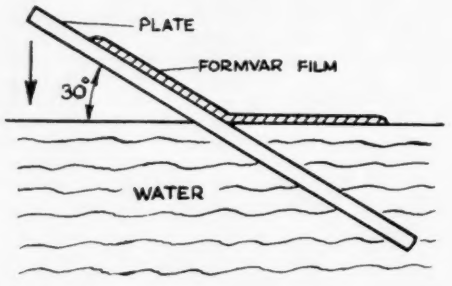
(I) by Transmission and (II) by Reflection.

In the former case the electron beam is made to penetrate a very thin layer of the specimen material which is normal to the beam, while in the latter case the beam is directed at a flat surface of the specimen at grazing incidence and undergoes Bragg-reflection in the surface layers. The term ' reflection ' is also loosely applied to the case where the beam is directed on to a surface at grazing incidence but the pattern obtained is due to transmission through the tips of

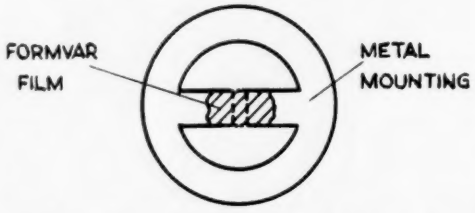
The Preparation of Formvar Films
(Germer)



A drop of Formvar solution in chloroform* is placed on a slightly greasy microscope slide and spread by the movement of a razor blade as shown. The thickness of the final film is determined by the clearance between blade and slide.



After the chloroform has evaporated the resulting film is floated off onto water.



The film is floated onto a metal mounting (preferably made from material such as copper-beryllium alloy) provided with a slit 0.05 mm. wide and lifted from the water. The diagram shows the mounting viewed from above and is larger than actual size.

* Formvar No. 1595 (Shawinigan Products Corporation, U.S.A.) or 'Resoglaz' dissolved in benzene may be used.

very small projections such as crystallites, rather than Bragg-reflection in the surface layers of an atomically smooth face. In this paper the term will be used in the looser sense, *i.e.* to cover all cases where the beam is at grazing incidence. Transmission usually gives clearer patterns with less diffuse scattering.

I. THE TRANSMISSION METHOD

The Specimen Support

Screens. The specimen is mounted upon a wire mesh or a suitably perforated wire plate, usually the former. The mesh should have a gauge of 30 wires p. cm. or finer and should be of a non-reactive metal such as pure nickel or platinum. It is freed from grease immediately before use by a brief heating at red heat in a Bunsen flame. Specimens which are themselves in the form of thin films may be supported directly on the mesh, but powders and similar deposits must be spread upon a thin film such as collodion which is mounted on the mesh. Cellulose-acetate, Formvar, etc., may be used also in the place of collodion.

Collodion Films. The film is made by allowing a drop of collodion to fall on to the surface of a beaker of clean water. The concentration of the collodion solution is adjusted so that one drop, after spreading and evaporation of the solvent, gives a film of the correct thickness. This is too thin for interference colours to appear—though these may be noticed while the solvent is evaporating—and the film will only be seen if suitable oblique illumination and a dark background are used. The thickness is tested by pushing a thin wire through a peripheral portion of the film and attempting to cut the film by moving the wire sideways. If the film puckers ahead of the wire it is too thick. If it breaks into small islands it is too thin. If the wire cuts cleanly and easily, it is of correct thickness. A piece of the film may then be taken by sliding the wire mesh beneath it and lifting gently. The edges of the mesh should cut the film as it is lifted, thus removing a section with the shape of the piece of wire mesh. Immediately before making a film for actual use, the water surface should be freed from dust and grease by spreading a rather thick film over it and then removing it completely.

Germer[1] gives details of the preparation of Formvar films, see Fig. 1, and of the evaporation of substances onto them in vacuum.

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The Preparation of Specimens from :—
(a) Powders, Suspensions, Smokes

Ideally, each particle of powder should be small enough for the electron beam to pass through it. Large particles not only obscure the beam but tend to become electrically charged and so distort the pattern obtained from any smaller adjacent particles. If a powder is too coarse for direct use it may be necessary to reduce it by ball-milling.

The number of particles per square centimetre of film should be sufficient to give a large number in the cross section of the electron beam, but not so dense as to obscure the beam. This can only be found by trial and error, but a rough rule is that a deposit such as graphite should be sufficiently dense to render the film translucent and grey, but not opaque when viewed against a light. Powders of transparent particles such as colourless crystals will not, of course, appear so optically dense as graphite at comparable particle concentrations. Aqueous suspensions are best applied to the wet (wire mesh) side of the collodion. The main bulk of the superfluous liquid is then removed by touching the edge of the gauze with a filter paper and the rest by freeze drying in the camera itself. Thin collodion films usually break up if allowed to dry completely in air but remain intact if freeze dried. Methods of preparation should therefore aim at leaving the film still moist when it is put into the camera.

Fine crystalline deposits may be made on collodion by applying suitable solutions to the wet side and allowing them to evaporate. The solutions must wet the surface, spread evenly and evaporate sufficiently rapidly to give crystals small enough to be penetrated by the electron beam. The rate of evaporation must not be too rapid, however, or the cooling so caused may result in condensation of atmospheric moisture on the specimen. A mixture of two solvents is often useful since the rate of evaporation may be adjusted by altering the proportion of the more volatile component. For example, cadmium iodide is better recrystallised from a mixture of water and alcohol than from water alone.

As illustrated in Figs. 2 and 3 colloidal graphite may be added to suspensions so that the pattern from the graphite flakes, which lie flat upon the film, appears together with that from the specimen and hence acts as a reference for calibration. In suitable cases fine powders may be examined by incorporating them with the collodion solution before making the film.

The Use of Graphite for the Calibration of Patterns

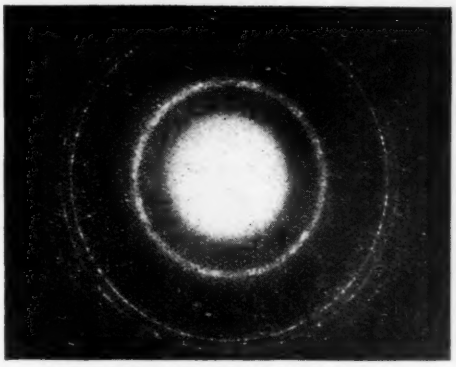

Fig. 2. Pattern from very fine graphite powder sprinkled onto a thin collodion film.

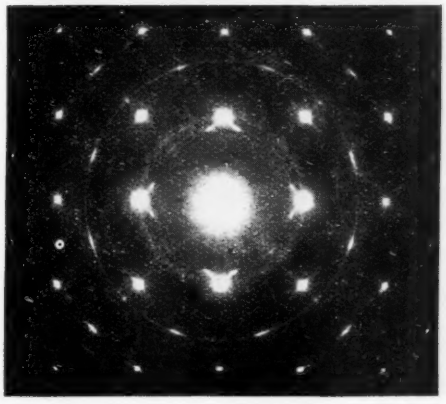

Fig. 3. Graphite is used as a reliable reference standard for the calibration of other patterns. For example, here the graphite rings can be seen together with the spot pattern from a (111) twinned Ag crystal film.

Evaporated metals, smokes and sublimates may be condensed onto collodion membranes as films or dusts for examination in the camera. Oriented crystallites have been obtained from smokes of molybdenum oxide and other metal oxides by Takagi[2]. The smoke was made by electrically heating a molybdenum wire in air and condensing it on a collodion film. The particles were platelike (0.1–1.0 μ in diameter) and when a drop of a suitable liquid was placed on the film and allowed to evaporate they became aligned with a common crystal axis perpendicular to the film. Similar results were obtained with chromium oxide formed by a d.c. discharge between chromium electrodes.

Identification of Hydrocarbons

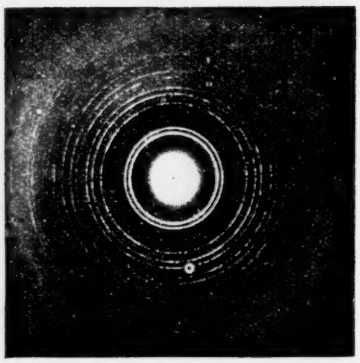

Fig. 4. Characteristic transmission pattern from a grease or long-chain hydrocarbon layer on a thin collodion film.

(b) Rubber, Oils, Polymers

Rubber films have been prepared for investigation[3] by spreading a few drops of solution in benzene on clean water in a Langmuir trough to give films initally 2,000 Å thick. These films were stretched to 5–10 times the initial length by drawing apart two chromium-plated bars placed across the trough in order to obtain partial orientation of the molecular chains. Parts of the stretched film were lifted off on nickel gauze and examined in the camera, no collodion support being necessary. Gutta-percha has been examined with a similar technique[4].

Films of polyvinyl alcohol have been prepared[5] by allowing a few drops of 0.2 % aqueous solution to fall upon a thin layer of celluloid laid on a suitable frame. After evaporation of the water the celluloid was dissolved in amyl acetate, leaving the film on the frame. The film was stretched for examination (100–200% extension) after exposure to water vapour and heating it in some cases. Other polymers, for instance methyl methacrylate[6], have been investigated by placing dilute solutions in acetone or benzene on water and allowing the solvent to evaporate. The resulting thin lenses were lifted from the surface on a mesh and examined by transmission. Storks[7] refers to gutta-percha and polyethylene acetate.

Oils which will form coherent thin films may be examined by spreading them over wire mesh or holes in metal plates (*e.g.* by dipping the plates in the oil). For example, silicones have been prepared[8] by wiping a drop of the liquid over a hole of 1 mm. diameter in a thin brass plate and evaporating the excess in the camera itself by making suitable arrangements to heat the specimen support.

Films of fatty acids[9] have been made by rubbing the fatty acid on to cadmium foil (which was previously degreased) and thinning the latter by floating it on ammonium nitrate solution (see Section (*c*)—Metal Films). Suitable pieces were picked up on silver gauze, washed by flotation on a clean water surface and examined in the camera. The pattern from cadmium and its oxide was present together with that from the fatty acid.

Hydrocarbons have been examined[10] by volatilising them onto a thin film of collodion 40–50Å thick, see Fig. 4. The resulting pattern was used to detect impurities.

(c) Metal Films

It is sometimes convenient to study corrosion, oxidation and similar reactions on metals by preparing the metal as a thin film through which electron transmission can take place and mounting it in the camera on a wire gauze similar to that used for

supporting collodion. For example, the course of oxidation by a corrosive atmosphere may be followed by observing the change in the electron diffraction pattern due to the build-up of oxidised layers after successive treatments of the metal film in the atmosphere concerned. Figs. 5 and 6 are illustrations of this.

Metal films are usually made either by thinning metal foil with an etching agent until parts of it become optically translucent or by evaporation of the metal on to a suitable surface in vacuum. In the former case the metal foil is floated on the surface of an etching solution. Suitable solutions are shown in the table below.

Table I.
Etching Solutions for the Thinning of Metal Foil

Solution	*Foil to Be Treated*
KCN–Solution	Gold, Silver, Copper
Hot KOH–Solution	Aluminium
HCL/HNO_3–Solution	Platinum
Dilute HCL–Solution	Tin

The precise conditions of temperature, concentration, pH etc., under which these solutions are used cannot be specified in advance but must be found by trial and error since small changes in constitution such as traces of impurity radically alter the rates of dissolution. When sufficiently thin, the metal is lifted off the solution with a wire mesh and washed by floating on pure, grease-free water.

The evaporation method of preparing a metal film for transmission is exemplified by silver. This is deposited on a cleavage face of rock salt in a conventional vacuum coating unit, or by evaporation from an electrically heated filament in an evacuated tube[11]. The cleavage face must be as perfect and free from steps as possible, otherwise the resulting film will disintegrate on handling. Immediately before inserting the rock salt slice into the evaporation unit the filament of the latter is freed from grease by heating so as to drive off a little of the metal onto the sides of the vacuum vessel. As soon as the filament is cool again the rock salt is inserted and the film deposited. The slice should be about 1–2 mm. thick and 0.5–1 cm^2 area. After receiving the metal

Study of Corrosion Phenomena

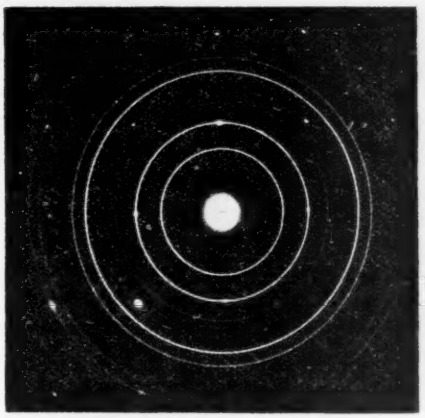

Fig. 5. Transmission pattern of a silver single-crystal film showing a square spot arrangement immediately after preparation. The film was subsequently exposed to the rapid attack of Br_2 vapour in air resulting in the formation of AgBr. Reaction products which have formed rapidly usually yield random polycrystalline layers giving rise to rings as shown.

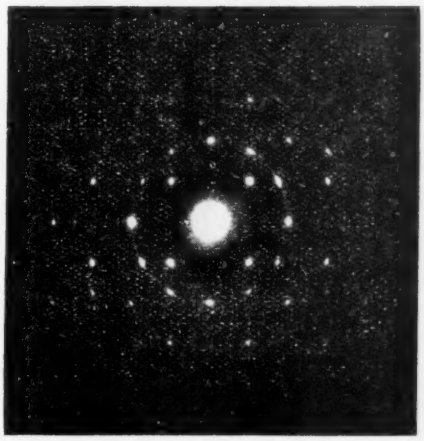

Fig. 6. In this case the silver film was attacked slowly by dilute Cl_2 in air. The slow formation of AgCl has resulted in its epitaxial growth in the form of strongly oriented crystals giving spots rather than rings.

it is laid (metal uppermost) on the wire mesh support, which is then set up so that the mesh is just in the surface of a trough of clean grease-free water. The same precautions have to be applied as for surface-film experiments in the Adam-Langmuir trough. As the underside of the rock salt dissolves away the metal film is lowered onto the mesh. It is washed by floating onto a clean water surface. The grain structure of the metal film obtained may be controlled by the temperature of the rock salt during deposition.

Typical Pattern of an Isolated, Unsupported Oxide Film

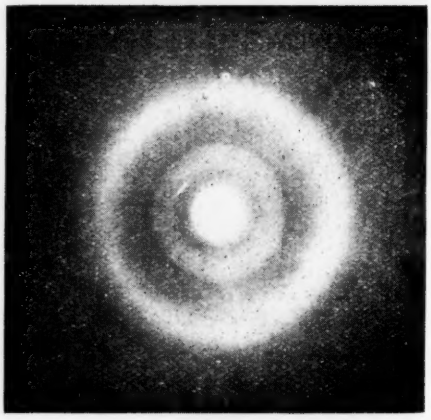

Fig. 7. Thin surface films on metals can often be stripped or else the metal removed chemically as indicated in the text on this page. They can then be examined by transmission as in this example of a mostly amorphous oxide film formed anodically on aluminium foil.

In the case of silver, if this is about 20°C. a random deposit is obtained, but if it is heated before insertion so that it remains at about 150°C. during deposition, the deposit is a mosaic of single crystals which have a cube axis parallel to that of the salt, but heavily twinned on $\{111\}$ planes.

Quarrell[12] prepared metal films by evaporation, electro-deposition and chemical reduction or displacement from solution.

(d) Oxide Films, Vitreous Substances

Metal oxide films may be examined by the transmission method if they can be removed from the underlying metal and if they are not too thick. The transmission pattern of an unsupported aluminium oxide film is shown in Fig. 7. For example, in a study of the passivation of iron[13], the oxide layer was detached from the surface electrolytically, using one of the methods proposed by U. R. Evans[14]. Other methods consist of scratching the surface and dissolving the underlying metal away with iodine or mercury. The electrolytic method has been further developed in connection with the preparation of oxide film replicas for the electron microscope[15].

Oxide films may also be used in a manner somewhat similar to collodion as supporting materials for specimens. Thus Boettcher[16] showed that aluminium films 250–300 Å thick deposited by vacuum evaporation on rock salt when heated in air at 600–900°C. produced an oxide, the cubic form of Al_2O_3, which proved useful as a carrier film for evaporated metallic multi-systems such as Al-Ag. The oxidation of thin deposits (*e.g.* of metals, silicon, etc.) is a general method of making oxide films.

Vitreous materials such as glass may be blown into thin membranes—too thin to give interference colours—by the usual methods of glassblowing[17].

II. THE REFLECTION METHOD

General Requirements re. Specimen and Specimen Support

In this method the ideal aimed at is a flat, smooth surface about 1 cm. in diameter, on which the electron beam can impinge at a grazing angle of 1° or less. Such surfaces may be prepared on materials to be investigated or may be used simply as flat substrates, on which to spread other substances.

In the investigation of abraded surfaces the clearest patterns are frequently obtained when the beam is perpendicular to the line of abrasion, since the electrons then have the best chance of passing through the tips of the crests which lie between the abrasion furrows. Films produced on smooth surfaces (*e.g.* by corrosion) are usually examined directly by re-

flection but if the corroded surface is roughened it may be advantageous to scrape a little of the product with a knife on to collodion or stainless steel and treat as a powder.

A suitable surface to act as a support for other substances may be made by carefully polishing stainless steel with fine alumina powder on selvyt cloth until trial in the camera shows that it gives a sharp shadow edge and only a little diffuse scattering. In some cases a cleavage face of good muscovite mica may be used as a highly smooth substrate, but this is a single crystal and may affect the orientation of some deposits. Clean glass, cut from microscope slides may also be used as a supporting surface[11]. If the deposit is prepared by evaporation, the substrate temperature may influence its nature as demonstrated in Figs. 8 and 9; the direction of the vapour beam may also be important.

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The Preparation of Specimens from :—
(a) Powders, Smokes, Condensed Vapours

The substance to be examined may be applied to a supporting surface in any appropriate way such as deposition from solution, sedimentation, sublimation, etc. For example, metal oxide smokes may be made by electrically heating a wire of the metal (*e.g.* molybdenum) in air, or by passing an arc discharge between electrodes of the metal. The smoke is condensed onto the polished stainless steel or glass surface by holding it a few centimetres above the wire or arc. Similar methods will suggest themselves for chlorides by using an atmosphere of chlorine, for instance. Powders may be applied conveniently by shaking them in a large bottle so that they form air-suspensions. The bottle is then opened and a little of the suspension allowed to pour out and settle on the specimen mount. Graphite may be added to a specimen in this way so that its pattern appears together with that of the substance to be examined, but the excess must be removed from the surface by gentle blowing or the pattern from the underlying material will be obscured.

Powders may sometimes be compressed into briquettes which are then prepared for the camera in the same way as compact solids, *i.e.*, by making flat

The Influence of the Substrate Temperature

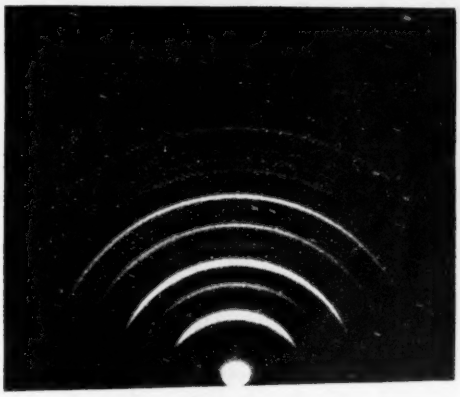
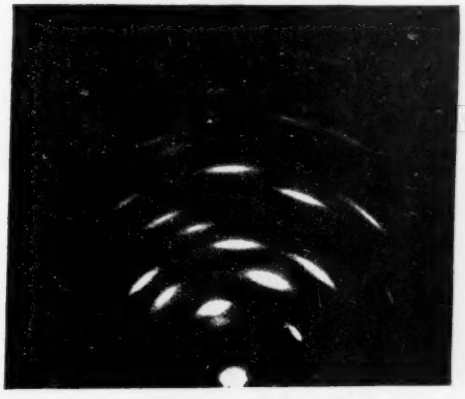


Fig. 8. Deposits of high melting materials from the vapour in vacuum onto substrates at about room temperature consist of random, relatively small crystals, as can be seen in this pattern of iron on glass.

Fig. 9. When the temperature of the receiving surface is raised, preferred orientation occurs : Compare this pattern of Fe on glass with that in the above Figure. This may happen inadvertently when a thick layer is being deposited and the receiving surface warms up due to the radiation from the evaporation source. Care must be taken either to avoid this condition or to take it into account when interpreting the results. The direction of the vapour beam affects the orientation in such cases and if it is not normal to the surface an oblique orientation axis develops.



surfaces upon them.

(b) Oils, Greases, Fatty Acids

These may be applied as smears with filter paper and in some cases molecular chains may be partially oriented by unidirectional stroking with filter paper or some other non-contaminating wiper. Such specimens often give the clearest patterns when the electron beam is perpendicular to the direction of stroking, since it can then penetrate the crests of the fine ridges which result from this method of application.

The Building-Up of Long-Chain Fatty Acids Multi-Layers *(Blodgett)*

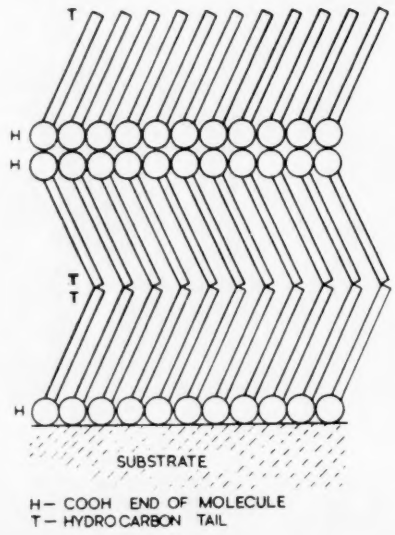


Fig. 10. Diagrammatic representation of substrate with three molecular layers of fatty acid upon it. The circles represent the –COOH groups and the straight portions the hydrocarbon chains.

Oriented films of long-chain fatty acids may be built up on a carrier surface, such as a glass or metal plate, by spreading the fatty acid as a monolayer in an Adam-Langmuir trough and dipping the plate into and out of the trough. The conditions of pH required have been given by Blodgett[18]. As the plate descends through the surface it acquires a monolayer and as it ascends again another layer is added but the molecular axis of the second layer is reversed in direction so that the –COOH and hydrocarbon groups of successive layers are adjacent to each other and form the arrangement :—head-tail, tail-head, head-tail, etc., as shown in Fig. 10. Thus the number of layers applied is equal to the number of times the plate has traversed the surface.

Monolayers of stearic acid on a crystalline substrate such as a cleavage face of molybdenite have also been prepared in this way[19].

(c) Single Crystals of Metals

Electron diffraction is frequently used to investigate how the nature of a deposit is influenced by the substrate on which it is formed. For example, the orientations of the crystals of an electrodeposited metal coating may be related to those of the metal on which deposition takes place. In such cases, results are more easily interpreted if they are obtained from deposits on known faces of a single crystal of the metal concerned[20]. The preparation of a suitable crystal face, therefore, is the starting point of many investigations.

With some substances, crystal faces may be obtained directly by cleavage or growth from solution but in a large number of cases this is not possible. This is so with most metals and since their crystals are soft, easily deformed and difficult to work without distortion and since also they are used in a wide variety of investigations into effects such as electrodeposition, corrosion, mechanical abrasion and catalysis, the preparation of a metal crystal will serve as a useful example.

Suppose the crystal to be in the form of a rod-shaped ingot such as might be obtained by cooling from the melt. It is first etched with a suitable medium[21]. Etching breaks the surface up into a series of minute steps, the tops and sides of which are microfacets parallel to crystallographic planes of simple indices such as $\{001\}$ and $\{111\}$. The chemical constitution of the etching bath determines which

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particular set of planes predominates. For example, copper etched with a mixture of 40g.$CuCl_2$ per litre of concentrated HCl gives mainly $\{001\}$ microfacets, but with 20% $NH_4S_2O_8$ it gives mainly $\{011\}$ and $\{111\}$ microfacets. When a pencil of light is directed at the surface of an etched crystal the microfacets act as a multitude of small mirrors, and if such a crystal is rotated slowly in a beam of light a flash is observed whenever it comes into such a position that a set of microfacets can reflect light into the eye. The normal of the plane to which the microfacets concerned are parallel then bisects the angle between the incident and reflected light. This is illustrated in Fig. 11 where C represents the end of a crystal with microfacets, normal to directions CA and CB, shown greatly exaggerated. Rays directed parallel to DS are reflected severally by the microfacets parallel to SE, following paths similar to the track FGH in the diagram. Orientation by such reflections is facilitated by setting the crystal up with one end (preferably hemispherically in contour—or approximately so) at the centre of a glass or Perspex sphere[22]. The principle is shown in Fig. 12. The position at which a pencil of light enters the sphere is marked at D^1 with a chinagraph pencil and when the reflected beam has been found with the eye at E the point E^1 is marked. The pole of the plane to which the microfacets are parallel is given by the point A which is midway between D^1 and E^1. The poles of as many planes as possible are marked in this way and the orientation of the crystal deduced from their symmetry. The angle at which it is to be cut is then determined with respect to the reference marks on the specimen or its holder.

Before cutting it is often advantageous to embed the metal in a supporting material such as wax or plaster of Paris. It is then cut carefully with a jeweller's saw, using a suitable lubricant such as alcohol. It is necessary to work slowly to avoid heating and distortion. The sawn end of the rod is then ground flat on metallographic emery paper firmly backed by a plane surface such as plate glass and using lubricant copiously. It is advisable to keep the specimen embedded in the supporting medium during grinding since this prevents it from being subjected to excessive

The Preparation of Single Crystals I. Determining the Orientation of the Lattice Planes

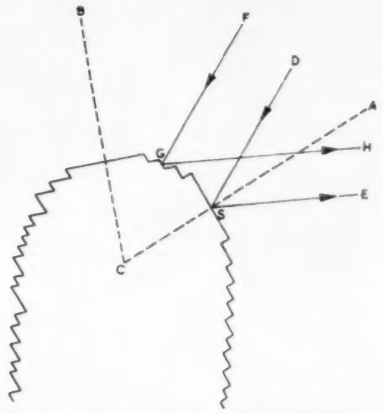
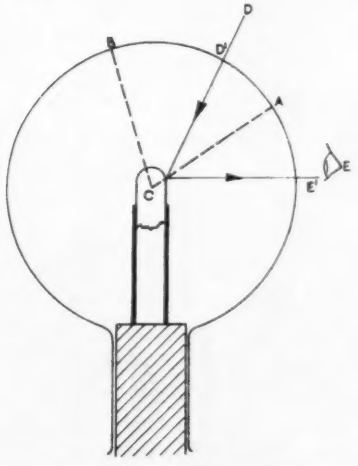


Fig. 11. Diagram showing the reflexion of light from an etched crystal. The etch microfacets, which in fact are microscopic, have been grossly exaggerated for visual clarity. The parallel rays FG and DS are reflected as the parallel rays GH and SE by the microfacets parallel to the set of planes to which CA is the normal.

Fig. 12. Orientation of a crystal by reflexion. A pencil of light DC is reflected along CE by a particular set of microfacets. The radius CA which bisects the angle DCE is the normal to the set of planes concerned and the pole A is marked upon the sphere. The poles A, B, etc., of all reflecting etch-planes are located and marked in this way.



pressure. Alternatively, it may be made to protrude very slightly through a hole, of approximately the same diameter as the rod, in a plate of the same metal. The rod is fixed to the back of the plate by a lump of wax such as picein, and the front of the plate together with the sawn end of the specimen is applied to the emery paper. The plate not only prevents the application of too much pressure to the specimen, but also minimises ' rocking ' during grinding, which

The Preparation of Single Crystals II. Location of Fixture for Cutting the Required Facet

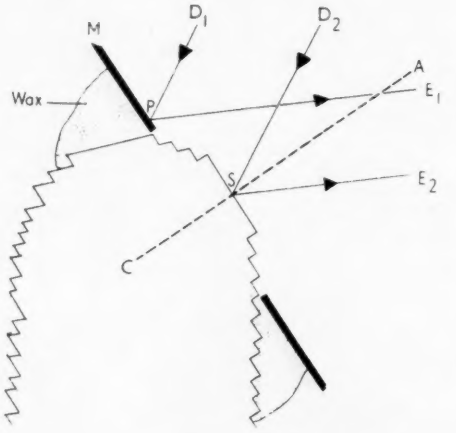


Fig. 13. Location of a metal plate parallel to a given set of etch planes. The wax is softened and the plate is moved until rays reflected from it such as D_1PE_1 remain parallel to rays reflected from the microfacets such as $D_2S\ E_2$. The wax is then hardened and the part of the crystal which protrudes through the plate is ground flush with it. (The microfacets are grossly exaggerated.)

leads to undesirable curvature of the surface produced.

If an etching agent is available which reveals the required crystal plane as an etch reflection, the metal plate may be set up parallel to this plane by manipulating it after the wax has been softened by gentle warming until the reflection of a distant light from it coincides with the etch reflection from the crystal[23]. This is illustrated in Fig. 13. A is the pole of the plane concerned. D_1P and D_2S are virtually parallel incident rays. When the plate M is normal to CA, *i.e.* when it is parallel to the reflecting microfacets, the reflected rays SE_2 and PE_1 are also parallel and the crystal then appears as part of the surface of the plate. The wax is then hardened by pouring water over it and the portion of the rod which protrudes through the plate is ground away, using the same technique as described below for a sawn crystal.

If the rod has been sawn, it is desirable to remove distorted material by grinding away about 1 mm. of the end with No. 1 or No. 0 emery paper before proceeding to finer grades and the rod may therefore be allowed to protrude through the plate to this extent. When this has been ground away gently and the plate is seen to be fully in contact with the paper and evenly abraded by it, the next finest grade of paper is applied working in a direction perpendicular to the first. This process is continued until the smoothest possible surface is obtained. The finest grades such as 0000 sometimes give trouble by embedding emery in the surface of soft metals, and when this occurs the surface is best left in the state reached in the preceding stage. As the grinding proceeds it is useful to examine the surface from time to time to see whether the material disturbed by sawing has been removed. This is done by spreading a drop of a suitable etching solution evenly over it. When sufficient etching has taken place and the surface has been washed and dried it should have an even appearance free from streaks parallel to the sawing direction.

When the surface has been ground as finely as possible there will remain a thin uppermost layer of mechanically distorted material. Wherever possible this should be removed by electropolishing[24]. If this cannot be done, etching is resorted to, but it should be emphasised that this should be as light as possible since a roughened surface scatters most of the electron beam instead of giving a clear pattern. It is best to etch so lightly as to give a barely perceptible change in the surface, then to examine the surface in the camera, and repeat the etch if necessary, until a clear pattern is obtained. When electropolishing is

carried out it should be borne in mind that some electropolishing baths leave a film on the surface which must subsequently be removed by treatment with, say, a dilute acid. For example, a satisfactory treatment for copper[25] after polishing in 50/50 phosphoric acid in water by volume is to wash first in distilled water then in dilute phosphoric acid and then in distilled water. Subsequently treat with 5% KCN solution for about 15 seconds to remove traces of oxide and then wash finally in distilled water. The crystal is immediately immersed in n-propyl alcohol to prevent contamination while being carried to the camera.

Removal of the metal by electropolishing is always preferable to mechanical working which is liable to distort the crystal. It is virtually impossible to work some crystals—zinc, for instance—without distorting them, and methods have been devised for shaping metals electrolytically, *e.g.* by shielding off all but a narrow band round the rod with a resistant tape and removing metal anodically from the unshielded area. Electrolytic jets in which the jet is made cathodic may also be employed[26].

Electropolishing of a specimen often leads to irregularities in the surface due to such factors as downward flow of the denser electrolyte which accumulates round the anode and it is then advisable to combine the mechanical and electrolytic methods by removing the bulk of the metal anodically and ' dressing ' the surface from time to time by grinding it flat on emery paper.

The author has devised an electrolytic buff for working flats onto rounded surfaces. The device has two important features : Firstly, the distance between anode and cathode is made as small as practicable so that the high points of the anodic specimen are appreciably nearer to the cathode than the depressions and hence more rapidly attacked, and secondly continuous mechanical removal of electrolytic products preferentially from the high points is brought about by rubbing the surface during electrolysis. This is illustrated in Fig. 14. The cathode is a copper dish with a flat bottom on which rests a layer of acid-resistant cloth such as woven glass fibre immersed in an electropolishing electrolyte. The specimen is the anode and is rubbed across the cloth with slow steady strokes. Comparatively high voltages and currents are used—say, 12V at 5A from a car battery — and considerable heat is dissipated necessitating water-cooling of the bottom of the dish. If possible the specimen also should be cooled. Failing that, it should be removed from the bath between strokes and the work should proceed slowly so that no overheating occurs. Electrolytic mixtures which are liable to explode, such as nitric acid/methanol or perchloric acid/acetic acid, must not be used.

The Preparation of Single Crystals III. Shaping of the Specimen by Electrolytic Buffing

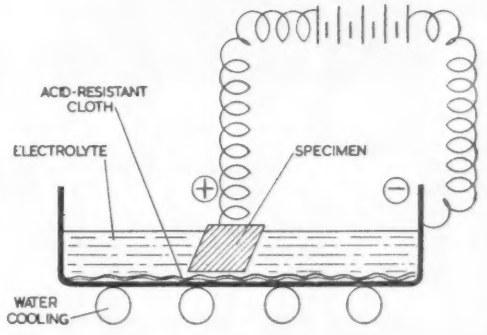


Fig. 14. The device shown was developed by the author and represents in effect an intensified and controlled method of electropolishing.

The method may be modified for polishing or shaping surfaces of a complicated design, too unwieldy to deal with in a conventional bath. Such a problem might arise in the investigation of corrosion in a working part of an engine when it might be desirable to electropolish the part to obtain a standard surface prior to its assembly into the engine and exposure to corrosion. In the modification the cathode consists of a copper tube through which cooling water is circulated and round which the acid-resistant cloth is wrapped and soaked in the electrolyte. The tube is

The Preparation of Single Crystals IV. Example of Definitions Obtainable with Well-Prepared Specimens

Fig. 15. Reflection pattern of electropolished (110) face of a copper single crystal prepared by careful sawing, abrasion and electropolishing as described on pages 145-146. Clear Kikuchi lines and few elongated spots show high perfection of crystal and an atomically smooth surface, indicating that the preparative methods used have not introduced distortions.

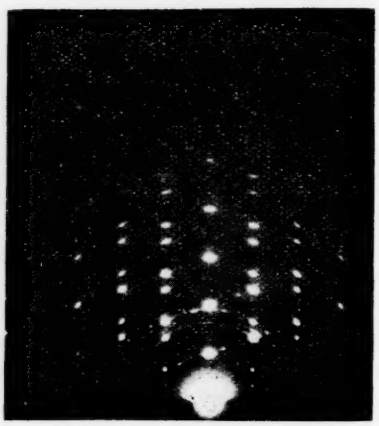


Fig. 16. The same crystal face after exposure to I_2 vapour at about 0.2 mm. Hg pressure and 305°C. The pattern from the underlying copper crystal has been superseded by that of CuI crystals which have grown epitaxially with (111) of the CuI parallel to (110) Cu and ⟨112⟩ CuI parallel to [110] Cu.

bent to a suitable shape and used rather in the manner of a paint brush. Thus it is possible to polish parts of the surface while leaving others, *e.g.* bearing holes, intact.

Methods analogous to electropolishing may be applied to crystals which will dissolve in a liquid. For example, instead of grinding, rock salt may be cut with a wet string. Any distorted surface material is then removed by very short immersion in a stream of water followed by rapid dehydration in absolute alcohol.

In the investigation of deposits formed (*e.g.* by oxidation) on a single crystal it is desirable to examine the structure of the thinnest possible deposit to determine how the initial deposit structure and orientation are influenced by those of the substrate crystal. Examination of thicker deposits reveals the structure at appropriate stages of growth. Even a layer a few atoms thick will obscure the pattern of the original crystal substrate and reference marks should be made on the specimen carrier to indicate its orientation so that the electron beam can be directed parallel to the main atom rows of the substrate surface at all stages of growth of the deposit.

(d) Polycrystalline Solids

In general the treatment of polycrystalline and vitreous solids is similar to that for single crystals but the treatment of substances containing more than one phase such as alloys requires special care since a particular etching agent usually attacks one phase more rapidly than another and the least attacked phase is then left standing in relief on the surface so that its pattern predominates. Heidenreich, Sturkey and Woods[27] laid down directions for the examination of secondary phases in alloys. The surface must be absolutely free from abrasion debris and corrosion products originating from the etching agents used. The latter must be selected to attack the major phase more rapidly so that the minor phases are left standing in relief. The most critical step is the rinsing immediately after etching. A treatment which proved satisfactory in the case of several alloys is shown in the table on the next page.

Table II. Method of Rinsing Alloys after Etching

Rinse I	50:50 methanol/acetone with 0.5% formic acid.
Rinse II	50 : 50 methanol/acetone.
Rinse III	Clean, dry benzene.
	Dry in blast of warm air.

The success of the rinsing procedure for several metals depends upon the use of rinses in which any metallic compounds formed are soluble and hence are not precipitated on to the surface. Solvents must be pure and freshly distilled. The authors discuss the factors which determine the choice of etchant. For example, it is important to avoid dissolution of a component and subsequent re-deposition on the surface in another form. Using these methods it is possible to identify and orientate 1.5% of a metal deposited in a single crystal matrix of another.

REACTIONS CARRIED OUT IN THE CAMERA

It is sometimes advantageous to arrange for deposition of a substance such as a metal film to be carried out in the camera itself by adapting one of the specimen ports to carry a tungsten or molybdenum filament from which the substance can be evaporated on to a suitable specimen holder. If it is desired to observe the diffraction pattern as the film is built up by this process, direct current must be used to heat the filament since the stray oscillating magnetic field from alternating current causes the electron beam to oscillate.

Reactions between specimens and gases may also be carried out *in situ* by admitting the appropriate gases to the camera with the specimen electrically heated, but in this case the beam cannot be maintained during the reaction. For example, Trillat and Oketani[28] prepared single crystal films of iron by condensation from the vapour onto rock salt cleavage faces at 600–700°C. followed by annealing at 700°C. After the rock salt had been dissolved away the films were examined by transmission, then heated in the camera in a mixture of carbon monoxide and hydrogen at 500°C. This led to the formation of

Artifacts Caused by:—
(a) Contamination

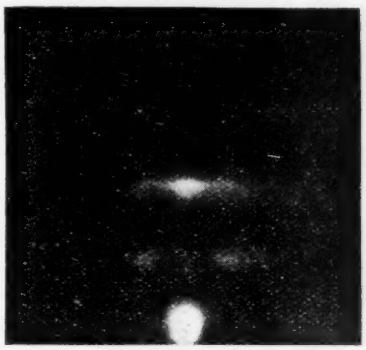


Fig. 17. Reflection pattern from grease which is a common surface contaminant (*e.g.* from fingerprints) but can easily be avoided with reasonable care and the use of grease-free solvents.

(b) Fragments of Embedded Abrasives

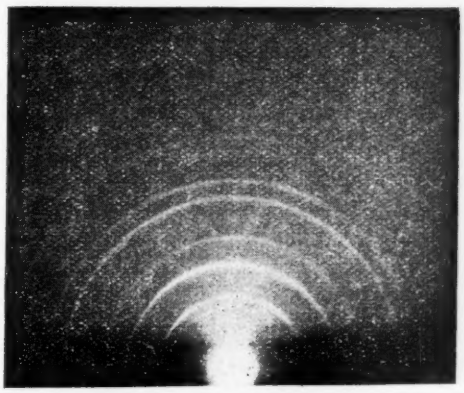


Fig. 18. Abraded or polished surfaces—especially, if no liquid lubricant has been used—often retain fragments of embedded material which may give a prominent pattern as in this example of a metal rubbed with MgO and then thoroughly washed. NaCl often deposited through handling with the fingers or as an impurity in chemicals gives a closely similar pattern which can be easily recognised.

cementite (Fe_3C) and its orientation relative to the original alpha-iron was determined.

Quarrell and Jackson[29] have devised a suitable high-temperature furnace for insertion into the camera. It consists of a steel sheath heated internally by an electric arc.

PRECAUTIONS

A layer of grease (see Fig. 17) or other impurity only a few Angström units thick will interfere with the pattern from a specimen and probably give rise to artifacts. It is important that the strictest care should be taken to degrease all objects likely to come into contact with the specimen and never to handle them with the fingers, which are a notorious source of grease, chlorides, etc. Filter paper fresh from a box which has been stored in a clean atmosphere is a fairly safe material with which to wipe surfaces and manipulate dry specimens. The use of cotton wool is not generally advised because unobserved strands often adhere to the specimen and become electrostatically charged in the camera. Abrasives, too, may cause artifacts by becoming embedded in a surface, particularly, if inadequate grinding lubricant is used. For an example see Fig. 18.

Many surfaces, especially metals, are rapidly contaminated by impurities in laboratory solvents and the atmosphere. The presence of halogens, hydrogen sulphide or sulphur dioxide, in the air of laboratories frequently leads to extensive reactions with metal specimens. Experiments should, therefore, be aimed at getting the specimen into the camera as rapidly as possible after preparation, say, within a few minutes or seconds. If possible the camera should be in a small room away from the laboratory atmosphere.

Non-polar solvents such as benzene usually leave traces of grease after evaporation while water, on the other hand, tends to promote oxidation of metal surfaces during drying. The final washing of metal specimens is, therefore, carried out with butyl or propyl alcohol, in which they are also kept immersed for protection during transit to the camera. All organic solvents are likely to contain sulphur or its compounds and only pure grades should be used.

Solvents such as carbon tetrachloride or chloroform may contain chlorine, particularly if they have been exposed to sunlight or oxidising conditions. Rubber bungs are a source of sulphurous impurities and should be avoided where possible. When their use is unavoidable they should first be boiled in dilute potassium hydroxide and then washed thoroughly before being allowed to come close to, or in contact with, specimens or solvents.

Systems which contain a volatile component such as loosely bound water of crystallisation cannot normally be studied in the camera.

When dealing with colloidal suspensions it should be borne in mind that they often contain appreciable quantities of protecting agents which may cause confusion in the results. For example, if the colloid particles are fairly large so that they obscure the beam, the pattern may be mainly that of the much thinner layers of other substances left when the liquid phase has evaporated.

Control experiments should be performed wherever possible. For example, before subjecting a crystal to surface deformation by abrasion it must first be ascertained that it is undistorted by obtaining a clear diffraction pattern from it. The control and the actual experiment should be performed on the same specimen since, especially in the case of single crystals, small differences in composition may have a relatively large effect on such factors as oxidation rate and mechanical properties.

The author wishes to thank Dr. H. Wilman for the very great assistance, he has given, in the preparation of this article and also for making available and personally preparing from the records of his original work all the photographs of diffraction patterns which appear in it. Thanks are due to Mr. C. R. Armstrong, B.Sc., for his assistance in developing the methods of electroshaping described. Messrs. Marglass Ltd. are thanked for their co-operation in selecting suitable acid-resistant cloths.

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a high temperature
RESISTANCE-HEATED HIGH VACUUM FURNACE

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Summary

MOST OF THE TECHNICAL FURNACES used for heating objects of reasonable size to high temperatures under very high vacuum are at present heated by high frequency induction, though the equipment is expensive and often difficult to obtain. An experimental 10kW vacuum furnace with a resistance heating element is described in this Report. It can be assembled from standard vacuum coating equipment to operate directly from the a.c. mains through a transformer. Promising operational results have been obtained and the design is suitable for the construction of larger furnaces on the same principle. The cost is about half that of an induction furnace of similar size and performance.

Sommaire

LA PLUPART DES FOURS INDUSTRIELS utilisés pour porter des objets de tailles normales à de hautes températures sous vide élévé sont chauffés par haute fréquence quoique l'appareillage soit cher et souvent difficulte à obtenir. Un four à vide expérimental de 10 kW. à resistance chauffante est décrit dans cet article. On peut le construire à partir de pièces standardes telles que celles utilisées dans un poste d'évaporation et l'employer directement sur le réseau alternatif au moyen d'un transformateur. On a ainsi obtenu des résultats prometteurs et on pourrait d'ailleurs le construire plus grand en partant du même principe. Son prix de revient est à peu près la moitié de celui d'un four à haute fréquence de la même taille et donnent les meme performances.

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INTRODUCTION

In recent years there has been considerable interest in the development of furnaces capable of heating solid objects on the laboratory and semi-technical scale to temperatures of 2,000°C and above under high vacuum. Such furnaces find application in the preparation and sintering of ceramic powders and metal-ceramic mixtures and they may also be used for the purification of refractory materials by vacuum sublimation of volatile impurities[1,2,8].

The methods of construction commonly employed incorporate either resistance or high frequency induction heating. The former usually requires only a transformer and regulator but the latter necessitates a special generator and control equipment. In both cases the current generated heats a conducting element which must withstand safely the desired operating temperature.

Resistance heated furnaces have usually been constructed from molybdenum or tungsten wire or rods with the heating element in close contact with a refractory support[3,4]. Providing certain precautions are taken this method is satisfactory for the attainment of high temperatures at ordinary pressures but is not so suitable for high vacuum operation owing to the difficulty of avoiding gaseous products from chemical interaction between the element and the refractory. Recently, attempts have been made to eliminate this interaction by freely suspending the heating element. These furnaces, however, have hitherto been very small in size [5,13] or intended for use at ordinary pressures[6]. In the design of larger vacuum furnaces by this method it has proved difficult to construct an element which will withstand the operating stresses and yet have an electrical resistance sufficiently high to facilitate power input.

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The rapid development of the vacuum high frequency induction furnace[2,7,9] is in part a consequence of these difficulties. The high frequency is best supplied from a motor generator set, for this permits very smooth regulation of the power input, a low working voltage and operation of the coil within a metal tank. The current lead-throughs into the vacuum tank are constructed concentrically or mounted in one insulation plate. They are required to carry a moderately high current since although the coil voltage may be 300 volt or more the power factor with respect to the coil is very low. The detailed design of the induction coil depends upon many factors[10] but there are no insuperable difficulties in the construction of relatively large units. Such units, however, are particularly expensive as they require, in addition to the furnace assembly, a source of high frequency and a complicated system of electrical control gear. It has therefore seemed desirable to examine the possibility of developing large resistance heated vacuum furnaces which can be operated directly from the a.c. mains through the relatively inexpensive variable voltage transformer. An experimental 10 kW resistance vacuum furnace of this type is described below. The operational results are sufficiently promising to suggest that larger units should be feasible and it is estimated that the cost would only be about 50% that of an induction furnace of similar size and performance.

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THE DESIGN OF VACUUM FURNACES

The following factors have to be considered in the design of a vacuum furnace:

- Size of the Hot Chamber.
- Method of Insulation.
- Heating Element.
- Power Supply.
- Size of the Vacuum Chamber.
- Vacuum Pumps.

The Size of the Hot Chamber

The size and disposition of the hot chamber is fixed mainly by the capacity and duty for which the furnace was required. A vertical cylindrical chamber, however, meets most laboratory requirements and facilitates construction of the heating element.

The Method of Insulation

The method of insulation and size of the chamber determine the power required to achieve the desired operating temperature. Insulation by radiation shields necessitates a relatively larger power supply but minimises the problem of outgassing and so reduces demand on pumping speed. It may also facilitate experimental or production work by increasing the maximum rates of heating and cooling which are possible. The efficiency of the radiation shields can be estimated providing the emissivity is known. In practice it has been found that with three molybdenum screens about 27 watt/sq. cm. is required at 2,300°C[7] and this is equivalent to about 17 watt/sq. cm. at 2,000°C since the energy required varies approximately as T^4 where T is the temperature in °abs.; 17 watt/sq. cm. is about 11% of the black body radiation at 2,000°C. By the use of a layer of refractory powder about 4 cm. thick the energy required can be reduced to about 5 watt/sq. cm. at 2,000°C. Zirconia, or thoria, if temperatures above 2,200°C are required, are suitable refractory insulating powders though in some circumstances carbon powder can be employed.

The Heating Element

The heating element will normally be constructed from molybdenum, tungsten, or tantalum though for special purposes certain borides and carbides are useful. Molybdenum is the most convenient up to about 2,200°C and it can be fabricated into heating elements by spot welding with thin tantalum inserts. At higher temperatures its lack of strength and relatively high vapour pressure make tantalum or tungsten preferable. The construction of the element depends upon the material used and the energy to be dissipated. With the refractory metals and at temperatures up to at least 2,200°C it is convenient to utilise a series of horizontal strips (as suggested by Dr. Pirani) in place of a single sheet for the element can then be constructed more robustly. Such an element can be further strengthened by equipotential cross-

members. It is desirable that at least one of the cross-members be extended to a firm fixing point outside the hottest zone of the furnace. The leads from the element may be clamped directly to standard water-cooled copper lead-throughs and the whole freely suspended in order to minimise difficulties due to thermal expansion. The element is held together by bolts, which can be in a relatively cool region, using suitable insulating bushes and rings.

The Power Supply

The total power required is determined by the consideration outlined above but since the element will in general operate at a maximum of only 30 volt in order to avoid arcing, a reasonable allowance must be made for transmission losses. There should be little difficulty in adjusting the arrangement of the element to suit the available voltage and the element can be constructed in sections if desired. With large chambers a symmetrical arrangement of three heating elements is possible thus permitting operation from a 3-phase transformer. Where conductor strips are used the cross sectional area is determined by the required resistance at the final operating temperature and is designed to match the characteristics of a suitable transformer. The latter should preferably have a wide voltage regulation in the primary for control purposes. The number of strips is determined by the total power to be dissipated and a check is necessary to ensure that the total surface area of the strip is sufficient to enable it to dissipate the required energy at a surface temperature not too far above that desired for the chamber.

The Size of the Vacuum Chamber

The size of the vacuum chamber is determined by the fact that the walls have to carry away the major proportion of the heat generated in the furnace. Efficient water cooling is required and a superficial area of at least 1,000 sq. cm./kW input is desirable.

The Vacuum Pumps

The pumping system required depends upon factors which are now well established in vacuum technology[12] and will not be discussed in detail here. If the high vacuum pump is suitably backed and separated from the furnace chamber by a line of negligible resistance the ultimate pressure is given by the following equation :

$$\text{Ultimate Pressure } (\mu) = \frac{\text{Rate of Pressure Increase with Pumps off } (\mu\text{l./sec.})}{\text{Speed of Pump (l./sec.)}}$$

The pressure increase in the furnace is a combination of the effect of leaks and the slow desorption of absorbed gas from the walls and insulation material. A total rate of less than 0.1 litre/sec. (preferably less than 0.01) is desirable for most applications of the furnace.

THE CONSTRUCTION OF A FURNACE

The construction of a 10 kW furnace designed to attain over 2,000°C will be described (See Fig. 1). The furnace was adapted from vacuum equipment originally supplied for use as a vacuum coating unit. It consisted of an 18 inch. diameter 30 inch. high vacuum bell exhausted by a nominal 300 l./sec. oil pump with a rotary fore pump having a speed of 224 l./min. Water cooling pipes were soldered to the bell and in order to accommodate the water-cooled electrical leads the normal base was specially modified as shown in the accompanying sketch (Fig. 2). The arrangement of leads used was similar to that which has been employed effectively for the introduction of the leads in high frequency furnaces. From the water-cooled copper leads the furnace element was suspended. This consisted of a series of horizontal strips of molybdenum welded to the main molybdenum connectors through 0.1 mm. thick tantalum inserts. Equipotential cross-member supports were provided and the method of construction is illustrated in Fig. 3. An alternative construction shown in Fig. 4 has also been successfully employed.

It will be observed that the main current leads were bolted to a radiation screen at about 3 inch. from each end of the hot zone using bushes and rings of recrystallised alumina for insulation. The lower bolt was not tightened and the molybdenum lead was slotted in order to permit differential expansion with

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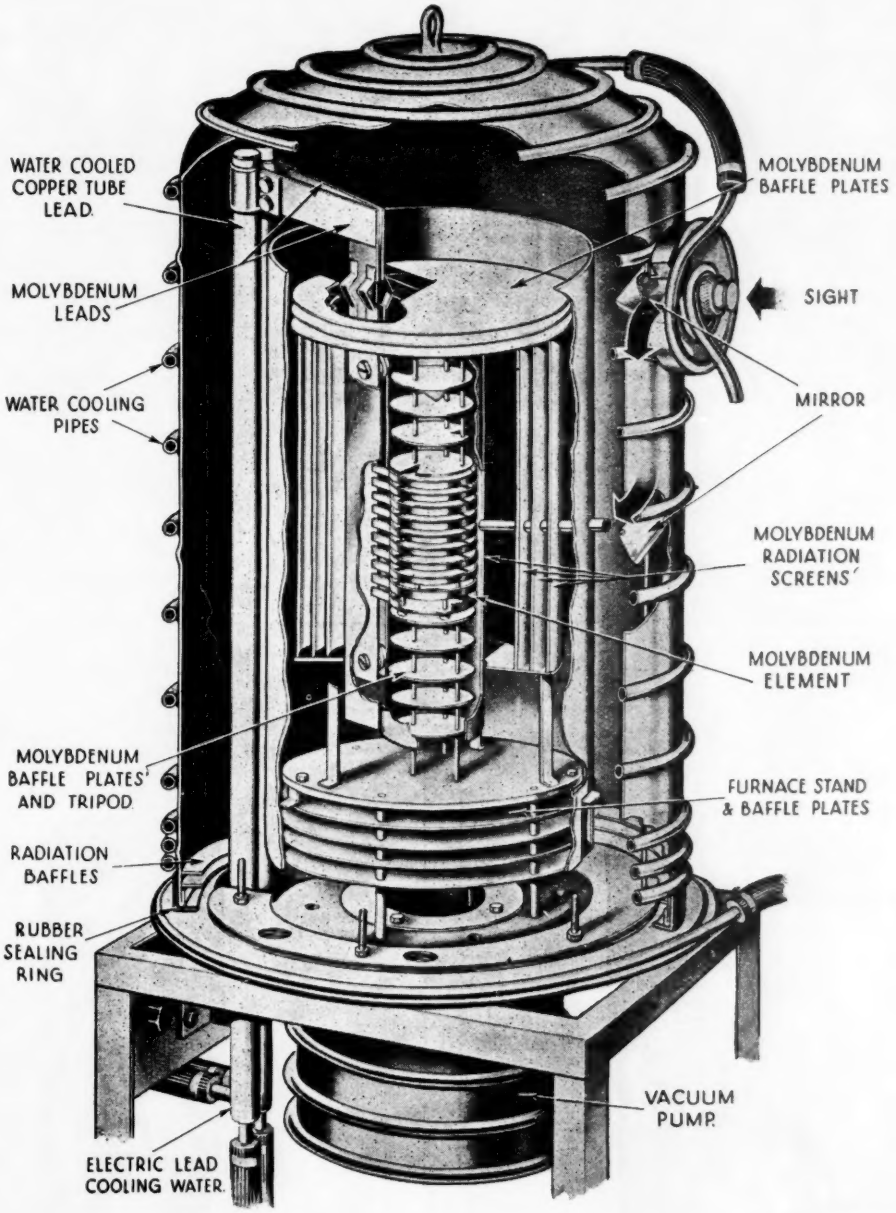

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Fig. 1. Molybdenum resistance furnace.

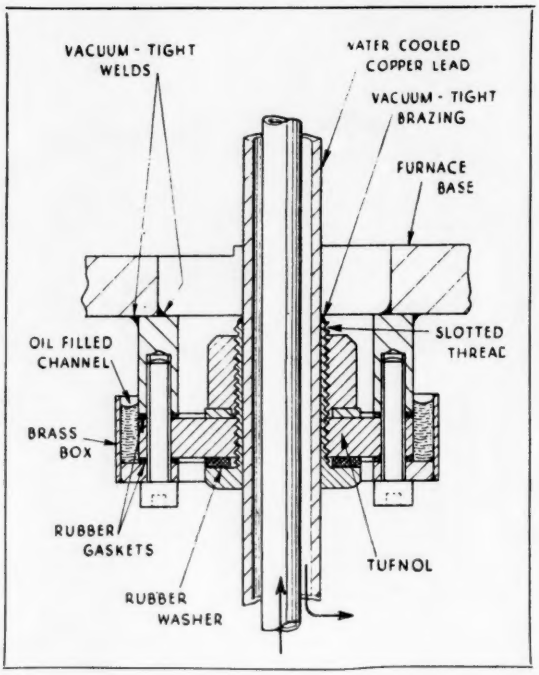
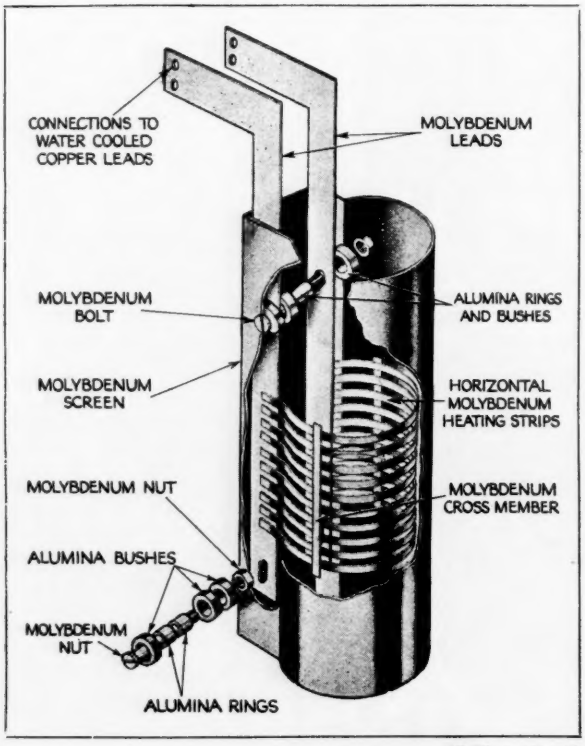
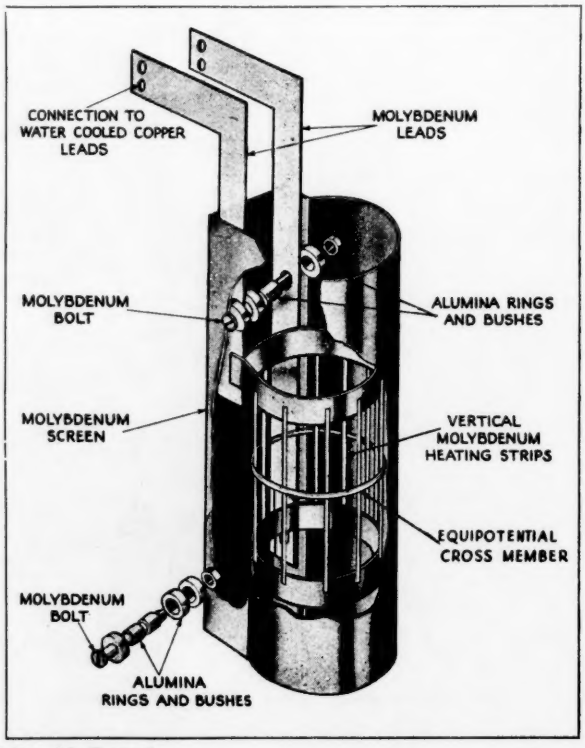


Fig. 2. Vacuum-tight lead-through base.

Fig. 3. Horizontal arrangement of heating strips.

Fig. 4. Vertical arrangement of heating strips.





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the outer shield. To avoid reducing the cross section of the molybdenum leads at the upper ends additional pieces of molybdenum were spot welded on each side of the holes. Four additional radiation shields were provided outside the first screen whilst a series of baffles served to provide insulation above and below the hot zone. Tungsten wool scrap was also inserted between two of the shields to provide additional insulation.

The available transformer gave a maximum energy of 12 kW at 12-18 volt. Allowing 2 volt for losses and for the use of radiation shield insulation this limited the size of the hot chamber to a surface area of 400-500 sq. cm. A hot chamber, 3 inch. diameter by 5 inch. high was chosen, this giving a surface area of 383 sq. cm. inside the first radiation shield which had an area of about 475 sq. cm. The effective length for each horizontal heating strip was about 22 cm. and 10 strips were employed. The resistance of molybdenum at 2,000°C was assumed to be 60×10^{-6} ohm/cm. To dissipate 10 kW at 10 volt each strip must have a maximum resistance of 1/10th ohm and a convenient cross sectional area was found to be 0.5×0.0275 cm.

The strips were spot welded at each end in three places to the main molybdenum leads using 0.1 mm. thick tantalum inserts. The leads were $1\frac{1}{4}$ inch. wide and constructed of molybdenum sheet 0.035 inch. thick. This gave a cross sectional area equal to twice the total provided by the strips. No special allowance was made for the resistance of the leads as this was difficult to estimate accurately and was provided for by basing the calculation on the lowest voltage available with the transformer.

The furnace was provided with an arrangement of mirrors to permit direct viewing of the hot zone from an existing window in the metal bell. Pressure readings were made by means of a standard combined thermocouple-ionisation gauge reading from 1 mm. to 10^{-7} mm. Hg.

THE PERFORMANCE OF THE FURNACE

The experimental furnace described has been operated successfully for a prolonged period at temperatures up to 1,800°C and occasionally up to 2,200°C. The operation has been either in high vacuum or in hydrogen at a pressure not exceeding 5 mm. Hg. Fig. 5 shows the relation between power consumption and temperature to obey an approximately fourth power law. The lowest temperature point was fixed by observation on the strip with an optical pyrometer whilst the others were obtained by the use of wires of known melting point. These were inserted inside the hot enclosure using pure alumina insulators. The melting of the wires was detected by the unbalance of a Wheatstone bridge network. A large resistance was inserted in series with the wire in one arm of the bridge and the heating effect was estimated to be negligible. The heat capacity of the furnace was of course relatively small and slow heating rates were used in the wire melting experiments. Nevertheless the equilibrium temperatures appeared to be slightly higher than recorded by this

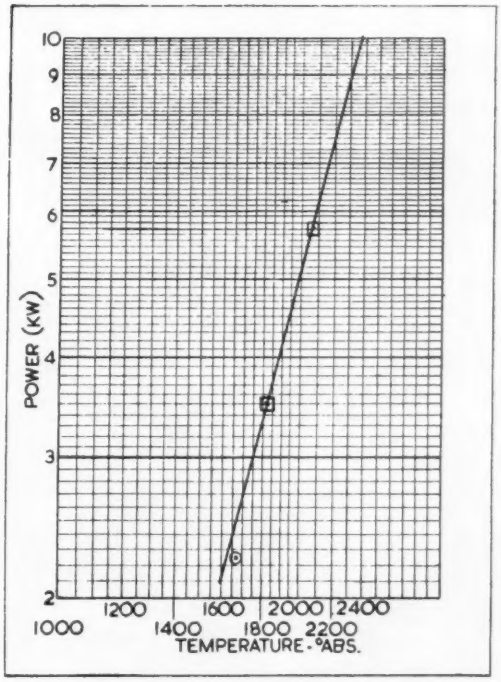

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Fig. 5. Relation between temperature and power supply to furnace.

method but the accuracy was considered sufficient for most uses of the furnace.

Measurement of cooling water temperatures showed that at 5 kW input the quantity of heat extracted from the bell was four times that taken out by the leads. The heat loss was therefore mainly by radiation, especially as some of the heat carried away by the copper leads would also have been transmitted by radiation. The power required to attain 2,000°C corresponds to 22 watt/sq. cm. calculated on the area enclosed inside the strips and horizontal baffles. This is larger than that reported for a high frequency furnace which had only three baffles[7]. These baffles, however, were more closely spaced whilst the hot chamber was completely enclosed. The surface area of the first radiation shield was appreciably larger (24%) than that bounded by the heating element as this was found necessary to allow for distortion of the heating element. At 1,700°C inside the heating strips a temperature of 1,550°C was measured by a probe wire between the element and first shield.

Some projecting pieces of the tantalum inserts have tended to become brittle under the conditions employed in the furnace but no effect on the operation of the element has been detected. Some indication of the performance of the furnace may be obtained from the results of an experiment on the sintering of a ceramic compact at 1,800°C(See Fig. 6). The fluctuations in pressure are due to the fact that the temperature was raised in steps. The furnace was always opened with an argon atmosphere inside to minimise outgassing difficulties. The rate of pressure increase in the furnace immediately before the commencement of an experiment with the pumps off usually indicated a 'leak' rate of the order of 0.2 μl./sec. At the conclusion, however, when the furnace had been thoroughly outgassed and was cold the rate of pressure change was considerably lower (approximately 0.02 μl./sec.). The total resistance was approximately 1/20th ohm at 2,000°C, 13.5 volt at the transformer giving a current of 625 ampere. This indicates that the total resistance of the molybdenum leads was about equal to that of the strips. No appreciable voltage loss could be detected in the leads from the transformer to the furnace. The thermal capacity of the furnace was small and rapid heating rates were possible though in practice the rate of increase in temperature was restricted so as to avoid undue stress on the heating element. The initial rate of cooling was also very rapid and articles were found to be quite cold after being left for

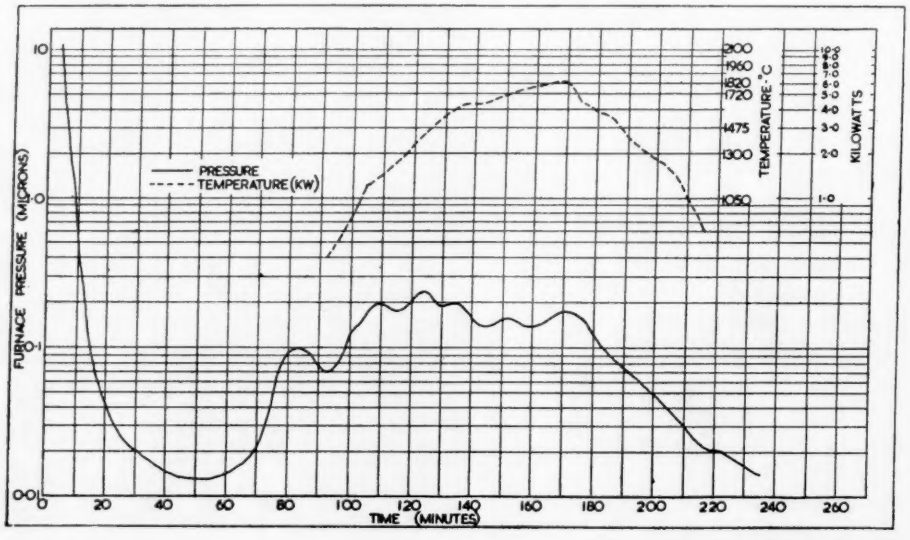

Fig. 6. Relation between furnace pressure and temperature during sintering experiments.

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four hours under vacuum.

The furnace has been operated for prolonged periods at temperatures up to about 1,800°C. At higher temperatures, however, and particularly at 2,000°C softening of molybdenum has caused difficulties due to short-circuiting arising from distortion of the elements. This distortion is most marked in the main leads where the current is largest and appears to be due to the repulsive force exerted between parallel conductors carrying large currents in opposite directions. The arrangement of strips shown in Fig. 4 greatly minimises the length of such parallel conductors in the hottest region where the molybdenum is soft and is also advantageous in that there is no tendency for the weight of the strips to cause a bending moment on the welds as in Fig. 3. In both designs equipotential cross members can be effectively used to prevent undue distortion of the actual heating strips. When using the furnace under conditions such that appreciable quantities of metallic vapour were volatilised some difficulty was experienced with short-circuiting due to deposition of metallic vapour on the insulators and for prolonged operation under such conditions it would be necessary to modify their location. No special difficulty has been found in the use of welded elements providing the welds are carefully made. The ' equipotential ' connecting pieces provide for any non-uniformity in the cross section of the heating strips and no special machining tolerances were fixed.

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CONCLUSIONS

A 10 kW high temperature high vacuum furnace with a robust molybdenum strip resistance-heating element can be constructed from readily available equipment.

Larger furnaces can be designed in a similar manner and would be considerably more economical than induction furnaces of similar size and performance.

This paper is published by permission of the Director of the Atomic Weapons Research Establishment, Aldermaston. Thanks are due to Dr. M. Pirani for valuable discussions and suggestions and to Mr. Cosgrove of the Osram Lamp Works for advice on the welding of molybdenum. The elements first employed were assembled by Mr. H. O. Willrich of the Ministry of Supply Armament Research Establishment and have since been fabricated for us by Messrs. Murex Ltd.

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LETTERS TO THE EDITOR

The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area

Sommaire

ON DÉCRIT LA PRÉPARATION des lames sémi-transparentes en dioxyde de titane pour des moniteurs de télévision à trois couleurs, par l'oxydation à chaud dans l'air de couches de titane et on commente sur leurs avantages par rapport aux lames sémi-transparentes dichroiques. On donne des détails sur l'appareillage utilisé pour obtenir des couches uniformes et on fait mention d'une méthode pour éviter les déformations de la surface pendant l'oxydation à chaud de grandes surfaces de verres.

In one well known type of colour television display system used for high-fidelity monitoring, there are two beam dividing mirrors which are arranged to combine into a single picture the primary colour images produced on three separate cathode ray tubes. For obtaining maximum brightness in the final picture, the ideal beam-splitters are a pair of dichroic mirrors having confluent spectral response characteristics. Such mirrors, composed of five or seven layers of alternately high and low refractive-index dielectric materials, have been made and used for this purpose. There is, however, the inherent disadvantage with a dichroic mirror that the spectral response is extremely sensitive to changes in the angle of incidence of the incident light especially at angles around 45°, which results in brightness and/or colour variation within the picture area. Apart from this the cost and practical difficulties involved in producing multi-layer dichroic coatings with the necessary permanence and uniformity over large areas, make the use of an alternative mirror coating desirable.

In a discussion on colour monitoring systems with Mr. C. B. B. Wood of the B.B.C. Research Department, it was proposed by us that a single layer film of the rutile modification of titanium dioxide would form a suitable beam dividing mirror. A TiO_2 film of the appropriate thickness has a neutral spectral response, reflects approximately 45 per cent. of the incident light and transmits the remainder, these characteristics changing very little with varying angle of incidence. While the light gathering efficiency of a pair of titanium dioxide beam-splitters is considerably less than that of a pair of dichroic mirrors, their comparative ease of production, durability and resistance to attack in humid or corrosive atmospheres makes their use in a three tube monitoring system more attractive.

Large titanium dioxide mirrors of up to 17 inch. × 13 inch. in surface area for use as beam-splitters in colour television receivers have been prepared in this laboratory and were first shown in the 1954 Physical Society Exhibition. Interest has been recently shown in these mirrors by various manufacturers concerned with colour television development and it is believed that a report dealing with the successful preparation of heat oxidised coatings on glass surfaces of large area would be of value.

The method of preparing TiO_2 films of the rutile modification has been recently described by Hass[1] and it is not intended to discuss the deposition technique in detail here. However, we have been preparing titanium dioxide coatings for some time and can confirm Hass' findings relating to the dependence of the metal oxide structure on the purity of the evaporated titanium deposit prior to heat oxidation in air. Thus we have also found that titanium evaporated in a poor vacuum ($\geqslant$0.1 micron Hg) produces TiO_2 films of low refractive index (shown by Hass to be the anatase modification) and that titanium evaporated in a good vacuum ($<$0.1 micron Hg) produces the high index rutile modification when heat oxidised.

The powerful gettering action of evaporated titanium, which may result in the deposition of a highly contaminated metal layer, can be used to advantage for lowering the gas pressure in an evaporation plant with a high ultimate pressure. Thus, if the titanium vapour source is covered with a shutter during the initial stages of evaporation then the gas pressure may be greatly decreased thus permitting the subsequent deposition of a pure titanium film. The plants used in the preparation of the mirror described here, namely, a plant with a 3 ft. diameter horizontal cylindrical chamber in which the mirror was supported in a vertical plane, and a specially constructed plant in which the mirror was supported in a horizontal plane in a 24 inch. diameter chamber, were both efficiently exhausted and gettering was not essential. In both evaporation plants the glasses were rotated during evaporation with the vapour source offset from the axis of rotation. The uniformity of the thickness of the evaporated film depends on the type and position of the vapour source with respect to the rotating mirror. This has been discussed in detail elsewhere[2]. In the

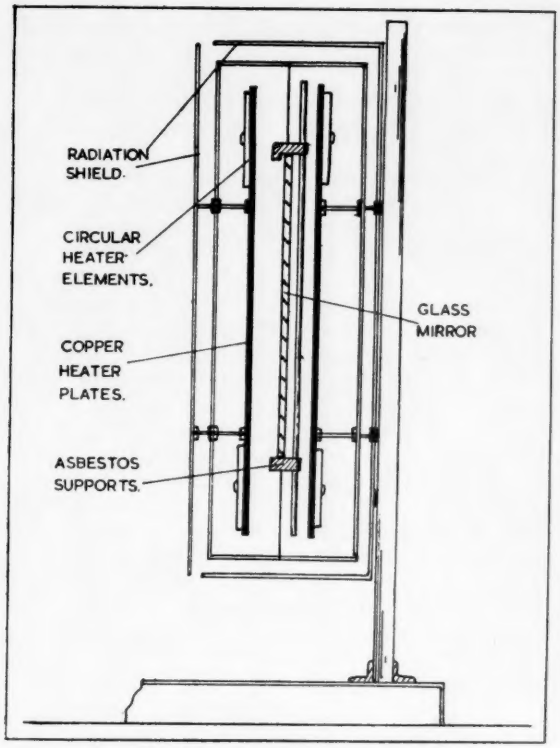

Fig. 2. Side view of vertical oven used in the heat oxidation of an evaporated titanium film.

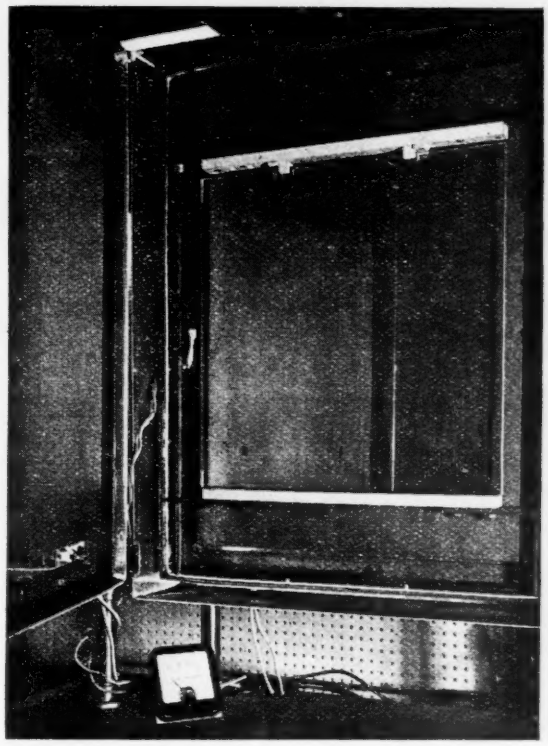

Fig. 3. Interior view of the same oven. One large copper heater panel can be seen on the inside of the open door and an oxidised high reflecting mirror clamped between the asbestos bars.

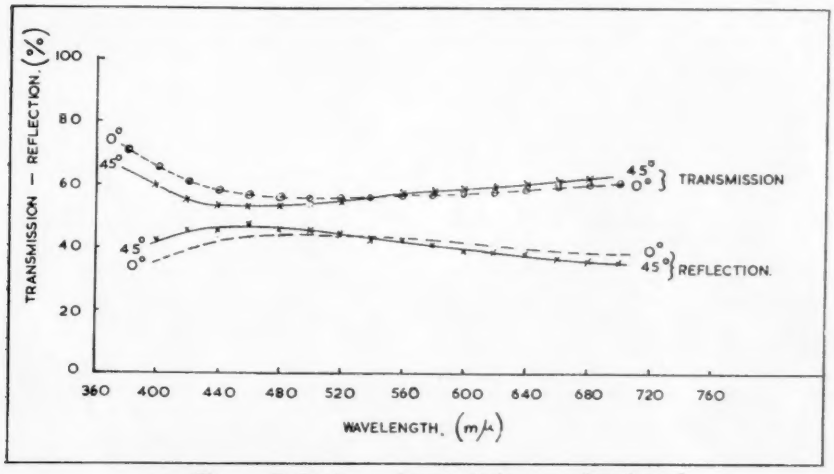

Fig. 1. Reflection and transmission as a function of wavelength of a TiO_2-film on glass (Ng 1.5) when measured at incidence angles of 0° and 45°. The TiO_2-film is 465 Å thick.

system with a vertical axis of rotation a tungsten spiral source heater was placed at a horizontal distance of 10 inch. from the rotation axis and at a vertical distance of 11.5 inch. from the plane of the mirror. With this geometry titanium films could be deposited on to glasses having a 20 inch. diagonal which, when finally heat oxidised, were sufficiently uniform as to exhibit hardly any variation in spectral response over the entire area.

The titanium metal was evaporated from a tungsten filament and before any coating was deposited the titanium was heated and completely outgassed. The glass or glasses were then introduced into the chamber and cleaned by subjecting them for a few minutes to a glow discharge. The evaporation rate was constant and fairly rapid, the operating pressure being less than 0.1 micron Hg. The evaporation was also continuous, since it was found that a break in the operation and then a continuation resulted in the formation of a contaminated titanium film which, when heat oxidised, had a low reflectance indicating an oxide other than that of pure rutile.

The film thickness was controlled during the deposition by measuring the light transmission of the growing titanium deposit using a photo-electric apparatus in which the monitoring light beam had been modulated to permit the amplification of the photocell signal. With the foregoing system, optical measurements could be made in normal daylight and in the presence of illumination from the vapour source. The design of the apparatus is to be described elsewhere.

Films of $\lambda/4$ thickness for $\lambda = 5{,}500$ Å were to be prepared. The titanium was evaporated until the light transmission of the metallic layer was reduced to 5 per cent. According to Hass a titanium film with this transmission produces a $\lambda/4$ thick film of titanium dioxide for $\lambda = 5{,}500$ Å when heat-oxidised in air. Fig. 1 shows the measured reflection and transmission *versus* wavelength curves of a TiO_2 film on glass at 45° incidence.

The evaporated titanium mirrors have to be heated in air to a temperature of 420° centigrade for at least two hours in order to oxidise the film completely. In early experiments the large mirrors were heated by laying them on a horizontal hotplate and covering their outer surface with an inverted tray. This method was found to introduce serious flatness distortion of the mirror surface in spite of the fact that the temperature was below the annealing temperature of the glass.

In order to ensure good registration of the three coloured images in a colour monitor, the mirrors must be perfectly flat, so that a better method of heating the mirrors had to be found. Possible reasons for the cause of the distortion due to the heat treatment were considered to be :

(*a*) unequal rates of temperature rise on both surfaces of the mirror producing internal strains.

(*b*) the mirror sagging or bowing under its own weight because the hotplate surface, on which the mirror rested, was either initially not flat or distorted during the heating cycle.

To avoid all of the foregoing possible effects a new oven was designed in which the temperature of the mirror could be raised uniformly and in which it was supported in a vertical position during heat treatment, so that any tendency for the glass to flow would not result in lateral distortion. A sketch and photograph of the general arrangement is shown in Figs. 2 and 3 respectively. The heaters are two $\frac{1}{8}$ inch. thick copper plates both having 'Variac'-controlled heater elements operated at a power output of 1.5 kW. The resistance heater on each panel consists of four annular-plate 500 W. elements* placed at regular intervals to give a uniform temperature rise. The glass is held centrally between the heater plates by two asbestos bars and is raised in temperature by both radiation and convection heating. The asbestos supports have been made adjustable by allowing them to slide on vertical rods positioned one on each side of the glass, hence the oven can accommodate a range of different size glasses. The glass temperature is measured with a thermocouple in contact with the film surface. The oven is encased by an aluminium radiation shield to reduce heat energy losses and thus minimise the initial warming-up time. A complete heating and cooling cycle is accomplished in about six hours.

The final process in the preparation of these beam-splitting mirrors is the deposition of an anti-reflection layer of magnesium fluoride on the uncoated side of the mirror in order to minimise unwanted reflections from this surface.

Some of the large mirrors prepared using the vertical oven described above gave completely satisfactory registration of the three separate images when set up in a colour monitor, thereby demonstrating adequate flatness.

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Crawley, Sussex, England.

15th September, 1954

References

[1]HASS, G., *Vacuum*, **2**, (1952), 331.
[2]HOLLAND, L., and STECKELMACHER, W., *Vacuum*, **2**, (1952) 346.

* Manufactured by Geo. Bray & Co. Ltd., Leeds.

The Thoughts and Actions Leading to the Formation of the C.V.T.*

Sommaire

M. DURANT, LE PRÉSIDENT du sous-comité pour la publicité du C.V.T. donne ci-dessous un compte rendu détaillé des évenements qui ont conduits à la formation du Committee on Vacuum Technique. On se souviendra que nous avons donné nos commentaires, dans notre dernier numéro, sur ces récents développements en Amérique et l'effet qu'ils pourraient avoir sur le développement de notre profession dans les autres pays.

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INDIVIDUALS, business organizations, academic institutions and governmental laboratories engaged in the use of high vacuum techniques and processes have long been unhappily aware that the vacuum field is an orphan among industrial, scientific and trade groups. This astonishing fact has led many to the conclusion that some sort of more or less organised group having as a common interest high vacuum technology, would indeed be welcome. In June 1953 a group of some fifty scientists, engineers, production men and operators, representing all types of industry employing vacuum technology, as well as vacuum equipment manufacturers, met in New York City in an effort to determine how their common interests in vacuum technology might best be served despite their otherwise widely divergent acitvities. The idea of a formal technical society with all the attendant formalities and trappings was quickly abandoned since the majority present felt that there are already too many technical societies competing for a man's time.

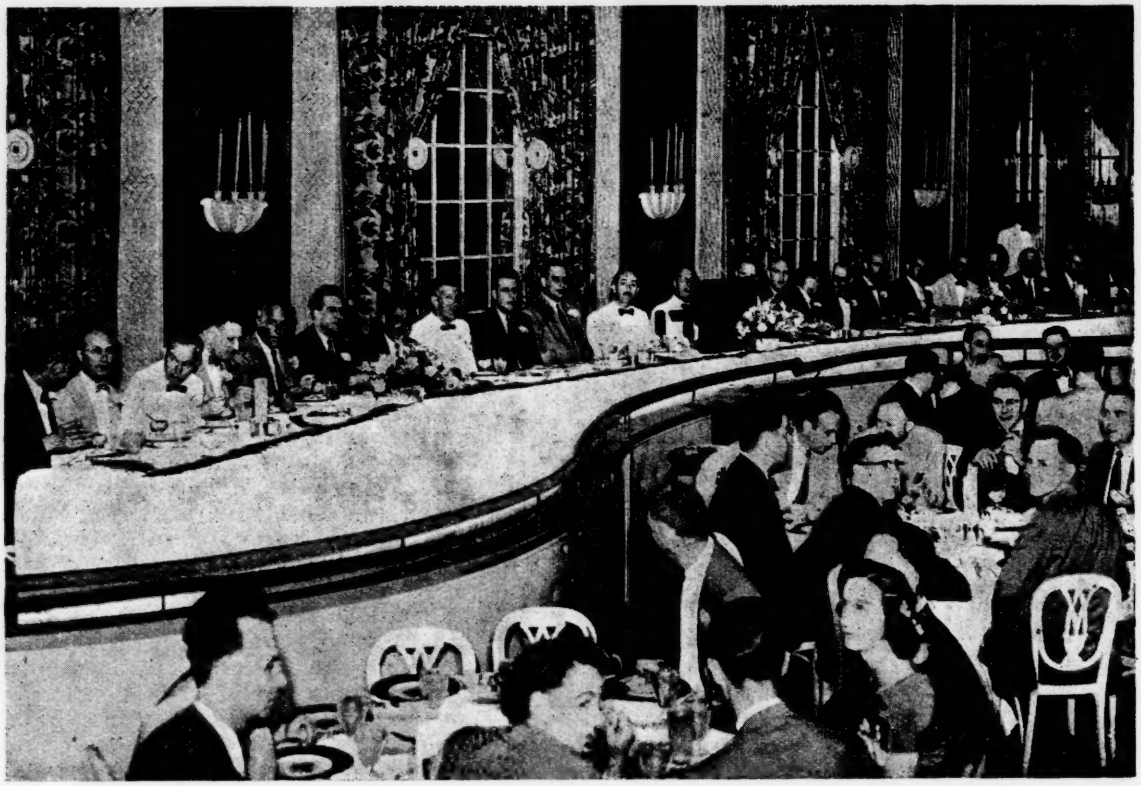
A review of the situation disclosed that while the literature dealing with the mathematics of vacuum and its theories is reasonably complete largely due to the impetus of nuclear research during the past decade —and information on equipment is readily available owing to the aggressive sales effort of the several manufacturers of excellent vacuum equipment— there was a large gap in the ' lore ' of high vacuum technology, or in the personal contact between workers in the field where one ordinarily has the opportunity to discuss problems of mutual interest. The group decided that this need could best be fulfilled by a High Vacuum Symposium which would, for example, give the electronic tube engineer confronted with the problem of exhausting a cathode ray tube some conception of the work being done in the application of high speed pumping systems to nuclear devices in a government laboratory—a place where the tube engineer would not ordinarily have access.

The possibility of affiliating with one of the established technical societies to gain administrative assistance, mailing lists, and the like, was considered and rejected on the basis that to be of broad appeal the vacuum symposium must not depend on an established professional society composed of chemists, physicists, or metallurgists, but instead must attract members of each and all of these professions—a virtually impossible assignment for a technical society identified with a single profession. Accordingly the Committee on Vacuum Techniques was founded on a non-profit charter to provide the administrative structure for the planning, promotion and execution of a symposium. Members of the committee were nominated and elected at the New York meeting. Committees on permanent organisation, finance, programming, standards, education, symposium planning and publicity were set up and charged immediately with the task of putting on a symposium in the summer of 1954. Emphasis was stressed on avoiding extremes which would tend to make the group either into a trade organisation—on one hand —or a ' long-haired ' scientific society on the other.

It is our belief that the Symposium held in June 1954 was successful. While it is never possible to achieve complete agreement among as many as 300 attendants, it was judged also that the decision to steer a middle road met with the approval of the majority. In the future symposia will be held as the amount of new work warranting discussion by means of papers accumulates.

One gratifying aspect of the Symposium was the great amount of interest shown by several workers in the field from overseas as well as attendance by several. It is felt that a great many more delegates from foreign countries would have attended if there had been more notice of meeting and wider publicity. It would appear from the demonstration of interest from other countries that due consideration should be

Editor's Note: In response to our comments in the last issue on the C.V.T. Symposium, which took place in June, 1954, the chairman of the sub-committee on publicity and publication of C.V.T., Mr. Durant, sent us this brief account on events leading up to the formation of C.V.T. in a personal letter.



C.V.T. members at the head table at the banquet include, reading from left to right : L. A. Backer, Central Sales and Manufacturing Co. and Chairman Committee on Arrangements; W. G. Mathieson, Sylvania Electric Products, Inc.; J. W. Skehan, Machlett Laboratories, Inc.; R. A. Denton, Optical Film Engineering Co.; A. J. Guthrie, U.S. Naval Radiological Defence Laboratory; B. B. Dayton, Consolidated Vacuum Corp. and Chairman, Committee on Nomenclature and Standards; E. W. Flosdorf, F. J. Stokes Machine Co.; H. Bliven, Vacuum-Electronic Engineering Co. and Chairman, Committee on Finance; J. R. Bowman, Mellon Institute; H. F. Colvin, Consolidated Engineering Corp.; A. S. D. Barrett (Guest Speaker), W. Edwards & Co. (London) Ltd.; K. C. D. Hickman, Honorary Symposium Chairman, Consolidated Vacuum Corp.; J. B. Merrill, High Vacuum Equipment Corp. and Chairman, Committee on Permanent Organisation; G. W. Oetjen, Leybold-Hochvakuum-Anlagen GmbH; W. B. Nottingham, Massachusetts Institute of Technology; R. A. Koehler, General Electric Co. and Chairman, Committee on Symposium Programme; H. C.Weingartner, National Research Corp.; F. M. Jenner, Consolidated Vacuum Corp.; R. L. Sylvester, Kinney Manufacturing Co.; J. H. Durant, National Research Corp. and Chairman, Committee on Publicity and Publication; H. W. Schleuning, Polytechnic Institute of Brooklyn; F. A. McNally, Jarrell-Ash Co. and Chairman, Committee on Education.

given to making the C.V.T. a truly international organisation with affiliated chapters abroad having nearly autonomous local administration but enjoying the advantages of wide distribution and receipt of publication of symposia.

The Committee on Vacuum Techniques emerged from the June 1954 Symposium much the wiser from its experience and with adequate finances to initiate plans immediately for its next meeting. New committee members were elected and already the group is planning for its next programme which will be held either in 1955 or 1956.

J. H. DURANT.

Chairman,
C'ttee, on Publicity & Publication,
Committee on Vacuum Techniques, Inc.,
P.O. Box 1282,
Boston 9, Mass. U.S.A.

21st September, 1954.

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BOOK REVIEWS

The Physics of Experimental Method. By H. J. J. Braddick. (Chapman and Hall Ltd., London, 1954). Pp. 424. Price £1 15s. 0d.

Many physicists graduate without any significant knowledge and appreciation of that art and science, experimentation, for the very good reason that they have no sensible instruction in the subject. So often the experimental work of a degree course is limited to the performance of a large number of well tried and established laboratory routines, which were at one time considered convenient to the study of certain physical principles. Unless promoted with enthusiasm and co-ordinated with instruction in experimentation, even an ideal selection of experiments can do little more than illustrate a small fraction of the more important of these principles and acquaint the student with the common physical instruments.

In some schools of physics, the student is encouraged to regard each experiment, however well tried and proved, as a problem or an investigation, to be tackled on his own initiative. The critical faculties are exercised in the design of the experiment, the choice and construction of the apparatus, and the analysis of his measurements. The early part of his course provides training and practice in the very useful laboratory arts of metal work and glass blowing. With such an apprenticeship, the elements of statistical and practical mathematics are integrated with the laboratory work, but actual instruction in the subject of experimentation seldom forms part of the official curriculum, and its degree of dissemination usually depends upon the teachers responsible for the supervision of the laboratories. At Manchester University, special attention is paid to the rôle of experiments in the training of the physics student, and a lecture course entitled 'The Physics of Experimental Method', forms a recognised part of the degree course. It is this which gives the title to Dr. Braddick's book.

Parts of this book have a resemblance to the well established and used 'Modern Physical Laboratory Practice' by Strong *et al.*, but Braddick makes no attempt to detail the laboratory arts and manipulative procedures of experimental physics. As the title of the book suggests, the emphasis is placed on such aspects as uses, applicabilities and limitations of important instruments and techniques in experiments. For the most part these two texts are complementary despite the apparent similarities of some sections.

The book opens with an excellent chapter on the statistical analysis of errors and the reduction of observations. An account of some useful mathematical techniques for handling experimental data includes some homely advice on the use of slide rules, logarithm tables and calculating machines. This common touch, in evidence so much throughout the book, adds to its attraction. The one regrettable omission here is the mention of, or reference to, the design of experiments after the manner of Professor R. A. Fisher.

The important principles of kinematic design receive due attention prior to considerations of the design and construction of instruments and apparata. To assist further the would-be designer, the physical properties of important materials like glasses, quartz, and the newly developed plastics and alloys are detailed and where possible conveniently tabulated.

The section on electronics forms a large part of the book, ranging from power supplies, through amplifiers, to counting circuits, with applications directed mainly at nuclear physics. This rapid scan of an extensive subject is not very satisfactory. The introductory part is too elementary to be compatible with the advanced character of the latter part, and many of the more novel and interesting circuits are referred to but not described in the text. The widespread applications of feedback theory to controllers, would appear to justify some expansion on this topic. The various types of noise which impose natural limits to electrical measurements are discussed at length, and valuable practical examples in amplifier design illustrate the magnitudes of effects due to fluctuation phenomena.

The survey of electrical measurements covers in detail just a few topics, such as the measurement of small currents and the design of the moving coil galvanometer. Whilst it is understandable that much must be missing, surely diagrams and essential details of basic bridge and other measuring circuits could have been included, if only in tabulated form.

The chapter on vacuum technique is both inadequate and unbalanced. Space is wasted on such matters as general descriptions of some commonplace vacuum equipment, whilst specific details are often lacking. How much more likely it is that the student will require to know of a simple control and measuring

circuit for an ion gauge, than the alleged ultimate pressures attainable with various not very common pumping fluids. Twenty five pages are not enough to do justice to the subject, but a synopsis of the main principles, techniques etc., might well have been produced by the more generous use of tables and diagrams.

Optical systems are required in numerous physical experiments, and very properly the subject of optics and photography are well and comparatively extensively treated from the aspects of image formation and the control of illumination. Here there is very much of use and interest, which is unlikely to be found in so readable or accessible form elsewhere.

The final chapter details some techniques in nuclear physics and includes descriptions of electron and ion collection chambers, counters and ancillary electronic devices. This section like much of the rest of the book will not be wholly intelligible, nor detailed enough in its parts, for the uninitiated. However, the plentiful up-to-date bibliography should repair omissions and give further body to the skeleton of the book.

No doubt some readers will be disappointed to find no references to such matters as model and analogue experimental techniques, temperature measurement and control, high pressure and low temperature physics. Who but a reviewer can really cavil at Dr. Braddick's necessary limited selection of topics ? He very properly lays claim to draw the attention of students to some of the available techniques, with no intention of competing with more specialised books detailing particular procedures. As previously suggested, much of the book anticipates a mature knowledge of physics and by itself would not provide a suitable background text to a course in experimental physics, but for all that, it can contribute much to the teaching and understanding of the subject at the undergraduate level, and to the isolated research worker it should prove invaluable as a source of references.

No matter what criticisms may be levelled at this book, nothing can detract from its essential purpose to bring proper recognition to the study of experimental methods. Here is the result of an important pioneer effort which is a significant contribution to the literature of physics.

D. W. STOPS.

. . . *of vacuum interest*

Lectures

THE ENFIELD TECHNICAL COLLEGE, QUEENSWAY, ENFIELD announces a post-graduate course of 6 lectures on HIGH VACUUM TECHNIQUES, starting Monday, 11th October, 1954 from 7-9 p.m. The lectures will be given by A. Williams, B.Sc. (Hons.) A. Inst. P. The fee is £1 1s. 0d.

●

FURTHER LECTURES are announced as follows :

September 30th, 1954 at 7.15 p.m.
The Vacuum Dehydration of Foodstuffs.
Lecturers : Part I, Equipment—*J. M. Hay*; Part II, Application—*E. G. B. Gooding* and *E. J. Rolfe.*
Held at : Marischal College, Aberdeen.
Arranged by : Agriculture Group with Aberdeen Section, Food Group and the Scottish Branch of Institute of Biology.

November 3rd, 1954 at 7.15 p.m.
Vacuum Techniques with Particular Reference to Steel Tank Rectifiers.
Lecturer : *N. L. Potter.*
Held at : English Electric Co's. Lecture rooms, Salt Avenue/Sabine Street, Stafford.
Arranged by : Institute of Electrical Engineers, North Staffs. Sub-Centre Students' Section.

December 2nd, 1954 at 6.30 p.m.
Melting and Casting under Reduced Pressures.
Lecturer : *H. H. Scholefield.*
Held at : 4 Grosvenor Gardens, London, S.W.1.
Arranged by : London Local Section, Institute of Metals.

France. THE SOCIETE DE CHIMIE INDUSTRIELLE together with the Fédération Européene du Genie Chimique have organised jointly with the Société Française des Ingénieurs-Techniciens du Vide, a series of 'Journées Techniques de Paris' to take place concurrently with the third Salon de la Chimie early December 1954. One of the days was devoted to vacuum engineering and the following lectures were given :—1. The General Principles and Physics of Vacuum, by F. Prevot; 2. The Present-Day Physiognomy of Vacuum Engineering by D. Degras; 3. The Uses of Vacuum in Metallurgy by J. Stohr; 4. The Deposition of Thin Films in Vacuum by R. Henry; 5. Freeze Drying and Its Industrial Applications by J. M. Dunoyer. There was also a luncheon for all delegates attending the conference.

VACUUM
CLASSIFIED ABSTRACTS

Vol.
3
1953

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

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All abstracts published in this section are allocated to one or more of the subjects shown in the following list :

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28 — APPARATUS COMPONENTS
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Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

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10 — VACUUM ENGINEERING (GENERAL) — 10

Conference on High Vacuum Technique

69/I

Germany. Summaries are given of all lectures read to a conference on high vacuum techniques at Mainz in October, 1952. The following is a list of the papers and authors :—

A New Manometer for the Pressure Range of 10^{-3} Torr to Less than 10^{-6} Torr.
Author : *E. Weissmann.*
A metal version of the Klumb windmill-type Knudsen gauge was described. Outstanding features were magnetic damping and a bi-metal arresting device.

Evaporation Sources for High Vacuum Deposition Processes.
Author : *H. Gruber.*
Various crucible techniques and methods of heating the substance directly (*e.g.* by electron bombardment) were discussed and new materials of the refractory type suitable for evaporation sources were mentioned.

Experiences in the Field of High Vacuum Melting.
Author : *Winkler.*
The purpose of melting in a high vacuum was discussed and the advantages of induction heating mentioned.

Electrical Equipment Required for High Vacuum Melting Plant.
Author : *K. H. Brockmeier.*
Design and performance of induction heating equipment was discussed in detail.

A Method of Determining the Molecular Weight of Gases and Vapours in the Low Pressure Range.
Author : *H. Kollmannsperger.*
Details were given of an instrument for the determination of molecular weights which represents a combination of Klumb's Knudsen type manometer and a viscosity gauge developed by Timiriazeff.

The Production of a High Vacuum without Pumps.
Author : *D. Tauern.*
An attempt was reported to utilise the known properties of titanium of absorbing hydrogen at low temperatures and desorbing it at high temperatures in order to create a pumping effect.

The Viscosity Gauge and its Properties at Low Pressures.
Author : *O. Heilingbrunner.*
A new quartz fibre manometer which has an operational range of 10^{+3} to 10^{-4} Torr was described.

A New Method for the Machining of Thermionic Valve Components Made from Glass, Individually or in Small Batches.
Author : *F. A. Gladitz.*
Latest developments in machines suitable for the shaping of small glass parts and seals were described.

Control Devices for High Vacuum Plant.
Author : *F. Starzmann.*
Various control devices were described, a.o. a bi-metal manometer developed by Klumb and Hasse which could be used to actuate such apparatus.

Industrial Furnaces for High Vacuum Processing.
Author : *F. Regel.*
The advantages of carrying out metallurgical processes in vacuum and the technical requirements to make such processes successful were discussed.

Report by Anon.
Glas-und Hochvakuum Tech.
1, No. 8
160-163

Sommaire : Liste des conférences à la colloque sur la technique du vide poussé tenue à Mainz en Octobre 1952

Progress in High Vacuum Technique

70/I
Article by J. Kistemaker
Nederland. Tijdschr. Natuurk.
18, Oct. 1952
259-263

Holland. Recently published developments in vacuum pumps, high vacuum apparatus construction techniques, valves, controlled leaks, manometers, etc., are described briefly.

(*Nuclear Science Abstracts*)

Sommaire : Un article général sur les récents progrès dans la technique du vide.

The Design of High Vacuum Systems. Part I and II

71/I

United Kingdom. The article deals with the basic calculations required in the design of high vacuum systems to be used in chemical engineering and discusses the working principle and performance of rotary and diffusion pumps, traps and gauges from the practical point of view. In chemical applications the pressures required are normally higher than 10^{-5}mm. Hg and the volumes to be pumped consists usually of long tubulations. This calls for a careful design of the vacuum manifold. A high vacuum is defined by the concept of the mean free path, which indicates the average distance, a gas molecule travels between successive collisions. Its value, assuming air to be the gas present and a temperature of 20°C, is determined by $l_m = \frac{5}{P}$ cm., where P is the pressure measured in μ. Values of l_m for various gases over a pressure range from 10^{-7} to 10^{-1} are given in a graph. The conductance C of long circular pipes for the case of viscous flow (N_R smaller than 1,200) normally prevailing in the backing line, is given by Poiseuille's law and an empirical formula developed by Knudsen later, which for air at 20°C temperature becomes $C = \frac{12.1\ D^3 J}{L}$ l./sec. An equation and graphs are shown for the determination of J. By setting J equal to unity the same formula can be used for calculations in the molecular flow range ($D/l_m \ll 1$). In this flow region the conductance is directly proportional to the cube of the pipe diameter D and inversely proportional to its length L. Where a short length of pipe is used, the impedance of the entrance aperture has to be taken into account by multiplying the above formula by a factor α. Calculated and experimental values of α are given in a graph, plotted against L/D. The backing pump is usually a rotary mechanical pump operating on the principle of volumetric displacement. ' Speed ' curves for these pumps are shown in a graph. The development of the diffusion pump of the type now available was initiated by Gaede and continued by Langmuir. As a rule mercury diffusion pumps are used in chemical systems but silicon oil

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was found by the authors to be a satisfactory alternative pump fluid owing to its high oxidation resistance and low vapour pressure. The ' Ho coefficient ' representing the ratio of the observed exhaust speed to that of the theoretical case when the jet is replaced by a perfect vacuum serves as an assessment of pumps. Approximately 0.5 is a good value for pumps of about 50 to 100 l./sec. Various protective devices are described. Diffusion pumps should be earthed to protect the pump heaters against possible harmful effects of probing glassware connected to the pump with a Tesla coil. A control unit actuated by a breakdown of the water supply is described which makes use of a modified S.U. petrol pump. Finally, various forms of pressure-operated switches are mentioned to act when the vacuum fails. Cold traps constitute a form of condensation pump intended to improve the vacuum in the system by reducing the temperature of vapours present to a point where their pressure is less than the operational pressure aimed at by pumping. One important design factor is the operational temperature of the trap. Another is concerned with the desired effectiveness of the trap as a refrigerator. There are two principally different types of traps, those holding their own refrigerant and those requiring a Dewar flask. The former is more economical in consumption of the refrigerant. In conclusion, some practical advice is given on the operation of vacuum gauges, in particular, discharge tubes, Mcleod gauges and ionisation gauges.

Article by E. A. Bunt & R. J. McCulloch *Industr. Chem.* Oct. 1952 460-465 and Nov. 1952 503-508

Sommaire : On donne des conseils aux ingenieurs chimistes sur les besoins essentiels pour la construction d'appareillage à vide.

Development of a Large, Linear Jet, Mercury Diffusion Pump Having High Pumping Speeds in the 10^{-6}mm. Mercury Absolute Pressure Range 72/I

See Abstract No. : 41/II

A Simple Air Ejector as a Suction Motor for Dry-Boxes 73/I

See Abstract No. : 45/II

A New Design of Filter for Dry-Boxes. Type D/47 74/I

See Abstract No. : 46/II

The Use of Getters for the Production of Very High Vacua 75/I

United Kingdom. The pressure in a sealed-off tube can be determined from two rate values, the rate at which gas is evolved from the tube components, and the rate at which this gas is taken up by the getter. Therefore knowledge of the gettering process should be combined with information on gas evolution. A brief survey of the present state of this information is given, showing *inter alia* that carbon oxides constitute a major part of the gases evolving in electronic tubes. Concerning the gettering process, the reaction between barium and carbon monoxide is investigated in detail. During exposure to carbon monoxide the gettering rate decays rapidly from a few l./sec. to very near zero, thus limiting the capacity of the getter to a fraction of a l $l\mu/cm^2$. This decay which is not indicated by a change in appearance of the getter makes the getter inefficient for other gases as well. The decay is thought to be due to formation of a protective surface film whose molecular volume is larger than the atomic volume of barium. The influence of flashing conditions and getter temperature on the decay is studied. It is shown that the gettering of carbon monoxide is irreversible between room temperature and 300°C. A new property of getters made of thorium powder and similar materials is discussed. Such materials can be used as ' Selective Getters ' which remove oxidising gases from the valve atmosphere but establish a constant pressure of hydrogen which can be controlled by varying the temperature of the getter and the quantity of hydrogen dissolved in it. Experimental evidence shows that such a hydrogen atmosphere at pressures of the order of 10^{-7} to 10^{-6}mm. Hg reduces the interface resistance of oxide cathodes by a factor ten. (*Author*)

Article by S. Wagener *Vacuum* 3, Jan. 1953 11-23

Sommaire : Revue des connaissances actuelles concernant les getters et l'étude des divers facteurs entre autre les propriétés des getters sélectifs.

The Assessment of the Quality of Zirconium Powder Intended for Use in the Production of Vacuum Tubes 76/I

France. Zirconium powder to be used as a getter in thermionic valves must be free from impurities which volatilise at the temperature of the production process, deposit in the form of a dark coat on the valve envelope and prolong unduly the time required for exhaustion. An assessment of the amount of volatiles contained in the zirconium powder by means of a chemical analysis was found to lack the accuracy required. A new method has been developed, described in this article. The material normally supplied suspended in methanol was placed in a desiccator which was evacuated by a water jet pump to remove all traces of water and alcohol. A sample of the dry material was filled into a crucible and weighed. The crucible was inserted into a silica tube and the tube evacuated. The end of the tube carrying the crucible was placed into a small resistor furnace and the sample heated to 400°C. for half an hour followed by another half hour at 1,000°C. Subsequently the crucible was removed and weighed again. The loss in weight expressed in % serves as a means of grading the

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quality of the zirconium. Samples supplied from four different sources have been tested by this method showing a weight loss of 10%, 20%, 40% and 45% respectively. The behaviour of these materials was checked during production. It was found that the material showing a 10% weight loss produced no deposit on the valve envelope, whereas the remaining three materials did and, in addition, lengthened excessively the time required for pumping.

Sommaire : Une méthode de contrôle de la qualité des ' getters au zirconium.

Article by H. Figour & H. Bonnel. *Le Vide* 8, March 1953 1305-1306

The Formation of Adsorbed Layers in Vacuum Chambers and Their Detection by Electrons 77/I

Germany. Adsorption phenomena in evacuated vessels are investigated. Adsorbed layers of gas are evident in metal vacuum apparatus and Drechsler and Hess have shown that the speed of their growth can serve as a measure of the quality of an operational vacuum. The nature and properties of adsorbed layers are discussed with particular reference to conditions in the electron microscope and diffraction camera. Adsorbed layers normally originate from water vapour and hydrocarbons present in the residual atmosphere, the latter as a result of the use of greased joints or rubber seals in the construction of plant.

Sommaire : Etude sur les phénoménes d'absorption dans les enceintes à vide.

Article by H. Koenig *Vacuum* 3, Jan. 1953 3-10

Evaluation of Vacuum Gasket Materials 78/I

See Abstract No. : 71 II

Sorbed Gases and High Vacuum Systems 79/I

United States. The evolution of adsorbed, absorbed and occluded gases from metals or glass can cause a virtual leak of some magnitude in a high vacuum system. During the casting of aluminium it was observed that as much as 17 cm^3. of gas, mainly hydrogen, may be trapped in 100 g. of the processed metal. Two experiments are briefly described. The first providing for an air pressure of 10 micron Hg on one side of a cast plate and atmospheric pressure on the other indicated a ' leakage ' at the rate of 1×10^{-7} $ft.^3/inch^2$., hr. This was solely due to the liberation of occluded or absorbed gases from the metal as could be proved by the second experiment where helium was used on the high pressure side and a helium leak detector on the opposite side. No helium was detected by the instrument confirming that no real leakage existed. The rate of evolution of gases was found to rise exponentially with increasing temperature

Sommaire : Etude sur le dégazage sous vide des piéces en aluminium coulé.

Note by Anon. *Nucleonics* 11, Feb. 1953 37

Rapid Graphical Procedure for Oven Design 80/I

United States. In order to reduce the time and effort normally required to finalise the design of ovens such as bake-out ovens for vacuum systems, a graphical procedure has been devised which permits a rapid estimation of the heating power required, and the operational outer surface temperature of an oven, if the size of the oven the nature and thickness of the structural materials and the operational temperature in the oven are known. The uses of the method are demonstrated by application to a practical case.

Sommaire : On décrit un procédé graphique permettant de déterminer rapidement la puissance de chauffe requise et la température des surfaces extérieures du four.

Article by A. I. Bennett Jr. *Vacuum* 3, Jan. 1953 43-46

Vacuum Bakeout Ovens 81/I

See Abstract No. : 61 II

A Controlled Gas Leak 82/I

United States. A Leak-in assembly is described which facilitates the controlled admission of gas at a rate ranging from 10,000 to 1 to a system which is continuously pumped at a pressure level of about 10^{-5}mm. Hg. The essential component consists of a rod made from porous porcelain of the type described by Hagstrum and Weinhart. The rod is situated in a container partly filled with mercury. By means of a magnetically operated plunger the level of the mercury can be raised or lowered as desired. Two such units, one with a large-diameter rod and one with a small-diameter rod are connected in series *via* a small reservoir fitted with a Pirani Gauge. A stopcock-controlled line between the gas bottle and system, by-passing the leak-in units is provided. In operation, the assembly is pumped down, the mercury level in the first unit lowered to expose the rod and the rod in the second unit left submerged. Then the tip of the gas bottle is broken off and the gas leaked through the first unit into the intermediate reservoir up to a pre-determined pressure. Then leak No. 1 is submerged and leak No. 2 exposed to the extent required for the desired rate of admission of the gas to the system. The arrangement allows the use of gas bottles filled to atmospheric pressure and ensures that any chosen leak rate is kept constant for several hours.

Sommaire : Description d'un dispositif permettant d'introduire une quantité dosée d'un gaz dans un systéme à vide pour une durée prolongée, la fuite étant construite en porcelaine poreuse.

Note by J. Morrison *Rev. Sci. Instrum.* 24, March 1953 230 231

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Bibliography of Industrial Drying

83/I

United Kingdom. A bibliography on the subject of industrial drying has been compiled and published by the Department of Scientific and Industrial Research which covers the period from 1924 to 1950. It brings up-to-date a collection of references on the same subject compiled by S. C. Chigson and presented by E. A. Allicott in the *Trans. I. Chem. E.*, **2**, 1924, 93-110 which recorded 500 publications of the period from 1900 to 1924. The new bibliography is compiled from over 800 publications and contains more than 4,500 references to articles and reports. In the majority of cases an abstract is added. Drying is defined as constituting the removal of moisture by evaporation, generally with the aid of heating appliances. Industrial processes such as evaporation and mechanical dewatering are excluded. A small number of references on the subject of drying of gases, air conditioning and heat transfer are included. The work is available in three volumes and consists of five sections as follows :

SECTION 1—PRINCIPLES OF DRYING

Fundamentals ; Hygromatic and Non-Hygromatic Instruments ; Moisture Determination ; Sorption ; Air Conditioning and Gas Drying.

SECTION 2—DRYING PROCESSES AND EQUIPMENT

Drying, General ; Equipment, General ; Control ; Efficiency ; Corrosion ; Cyclones ; Film and Drum Drying ; Electro-Endosmosis ; Freeze Concentration ; Freeze Drying (Drying by Sublimation) ; Heat Pump (Application of—) ; High Frequency (Dielectric) Heating ; Infra-Red (Radiant) Heating ; Mill Drying ; Packaging ; Pneumatic Drying ; Rotary ; Spray Drying ; Solar Heat (Use of —) ; Tray Drying ; Tunnel Drying ; Turbo Dryers ; Ultra-Sonics (Use of—) ; Vacuum Drying.

SECTION 3—AGRICULTURE

Grain ; Green Crops ; Hops ; Pyrethrin ; Tea ; Tobacco.

SECTION 4—FOOD

Food, General ; Foods, Various ; Baking ; Coffee Extract ; Egg ; Fish ; Fruit Juice ; Gluten ; Meat ; Milk ; Pectin ; Fruit ; Vegetables ; Potato ; Yeast.

SECTION 5—INDUSTRIAL MATERIALS

General ; Leather ; Timber ; Ceramics ; Foundry Cores and Moulds ; Coal ; Peat ; Paper and Board ; Textiles.

Bibliography compiled by *D.S.I.R.* Dec. 1951 published July 1952

Sommaire : Le D.S.I.R. a publié une bibliographie concernant le séchage en general, celle ci comprend la lyophilisation et le séchage sous vide.

81/I

Dehydrated Meat

See Abstract No. : 30/IV

55/I

Vacuum Deposition Becomes a Trade

See Abstract No. : 53/III

86/I

High Quality Plating in a Vacuum

See Abstract No. : 54/III

13 — ELECTRICAL SCIENCES — 13

87/I

The Electrical Breakdown of Air between Insulators

See Abstract No. : 32/IV

88/I

Electrical Breakdown in Vacuum

See Abstract No. : 31/IV

89/I

Electrical Discharges

United Kingdom. Recent developments in electrical discharges are reviewed mainly for static fields, high frequency fields, cold emission phenomena and space-charge effects in arcs. The Electrical breakdown of gases in static fields is first discussed at low pressures. This is explained satisfactorily by the generalised Townsend theory. It is shown that the breakdown mechanism is controlled by certain constants occurring in Townsend's empirical equation and measured for the growth of the ionisation current under conditions remote from electrical breakdown. The physical significance of these constants is therefore investigated. The first constant depends on the primary ionisation by electrons while the other constant depends on secondary processes occurring during the motion of electrons crossing the gap. Secondary processes of importance at low pressures are secondary emission of electrons from the cathode due to the incidence of positive ions, photons or excited atoms. The effective work function of the cathode surface was therefore investigated by condensing different materials on to it. It was shown that by altering the surface properties or the geometry of a cathode, the predominating secondary processes can be changed at will. For breakdown at high pressures on the other hand many experimenters thought that secondary processes played no part and special electron avalanche

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theories were developed to explain the observed effects. But careful recent work has shown that even at high pressures the same generalised Townsend equation still describes the growth of ionisation currents in uniform fields with the same interpretation of the constants in terms of primary and secondary processes and there was no evidence of the sudden appearance of any new spark-producing processes. Experiments in nitrogen indicated that cathode effects were the predominant secondary effects. After breakdown an arc was formed resulting in a space-charge conditioned discharge. Photo-ionisation as a secondary process in air was investigated recently and shown to be important in certain cases, but its exact significance in every case was difficult to predict as yet. The formative time lag was also investigated. Observations are again in agreement with analysis based on the same primary and secondary ionisation processes. Extreme conditions of pressure are important for high voltage insulation where their full advantage is not always realised because of the occurrence of field emission at high field strength which upsets Paschen's law. The low voltage discharges occurring with very small gap distances, *e.g.* during the closing of contacts in telephone relays, are similarly explained by an extraction mechanism. High frequency discharges with electric and magnetic excitation are described but only the former has been studied experimentally to any extent. At low frequencies and high pressures the discharge is basically the same as with static fields. The high frequency discharge discussed occurs when the transit times of the electrons are short compared with the period. Then breakdown occurs at much lower potentials reaching a minimum under certain conditions. This is due to the influence of the walls and depends on the pressure, frequency and linear dimensions of the discharge tube. Here, too, secondary processes are important as in the case of static fields. For large values of these parameters the motion of electrons is confined to the gas but recombination, attachment and diffusion away from the ionisation region are of importance. At very low pressures (mean free path equal to or larger than electrode separation) there is no pressure dependence on breakdown and only secondary emission from the walls due to electron bombardment every half cycle explains the discharge mechanism. The experimental work and current theories of the high frequency glow and recombination and diffusion processes occurring at higher pressures and frequencies are discussed. Also cold electron emission and the associated failure of Paschen's law are discussed. Experimental investigations involving the method of mean statistical time lag were carried out to determine the dependence of electron emission on the electric field. Much increased emission was found for oxidised or contaminated surfaces and the mechanism was shown to depend on the thickness of these oxide films. The effect of space-charge once the discharge has been established is discussed for the case of an arc. The theories and properties of the arc column, the appearance of hot spots on the cathode and heavy current arcs as well as the processes involved in short arcs at the closing and opening of electrical contacts are reviewed. Finally, work on the nature of radio frequency plasma oscillations and the generation of noise by discharges is described.

Sommaire : Article général sur les décharges électriques dans lequel on essaie de donner une théorie de base expliquant tous les phénomènes déjà connus.

Paper by
F. Llewellyn Jones
Rep. Progr. Phys.
16, 1953
216-265

A New Electron Multiplication Process

90/I

United Kingdom. The mechanism whereby electrons moving through a gas in an electric field multiply their numbers as a result of collisions with the gas present is known as the Townsend avalanche. Another mechanism of multiplication has been observed in high frequency electric fields at very low pressures where secondary electrons are released as a result of the initial electrons hitting the walls of the vessel. This wall multiplication process was investigated by the authors. They used a cylindrical glass vessel fitted with external electrodes and filled with neon for their experiments. At frequencies equal to or less than 10^7c./sec. and pressures higher than 50mm. Hg the peak value of the starting field was found to correspond to X p = 0.6 V./cm. per mm. Hg. In this condition the electron energy is too small to cause multiplication by collisions. The alternative mechanism is explained as follows :—The first electron accelerated by the field will excite rather than ionise atoms it is colliding with and thus produce normal excited atoms emitting certain resonance quanta and metastable atoms. The latter will also emit resonance quanta on collision with neutral atoms but a few will hit the walls of the vessel and release there photo-electrons which will be accelerated, a process which could be termed a ' quantum avalanche '. In order to produce multiplication by this process the initial electron must produce a sufficient number of quanta to release more than one electron from the wall. According to calculations by the authors the suggested mechanism is likely to occur in the frequency range of 10^7 — 50c./sec. Once a sufficient number of electrons has been produced in this manner the ionisation of the gas will proceed by the Townsend mechanism.

Sommaire : On a étudié un phenomène nouveau, reconnu récemment, c.a.d. la multiplication des électrones aux parois d'une enceinte, qui se passe dans des décharges à haute fréquence.

Letter by
W. L. Harries &
A. Von Engel
Nature
171, 21.3.53
517

Methods of Increasing the Electrical Conductivity of Surfaces

91/I

United Kingdom. The generation of static electrification is a surface phenomenon. The rate at which a static charge is built up or dissipated depends on the nature of the surface. By applying a film of suitable material the resistance of the surface can be reduced effectively and dissipation of the charge promoted. For instance, a coat of semi-conducting glaze applied to a porcelain surface instead of the normal glaze reduces the surface resistivity from appr. 100,000 M Ω to a value between 0.2 and a few hundreds of megohms. The basic composition of the semi-conducting glaze is the same as that of ordinary glaze, but it contains a higher proportion of metallic oxides such as Fe_2O_3, ZnO, Cr_2O_3. Micrographs of ceramic glazes are reproduced. The resistivity of a glass surface can be reduced to a figure of 100 to a few 1,000 Ω by spraying it with tin chloride while it is hot which forms a transparent conducting film of stannic oxide on cooling or by depositing metallic oxide films with the help of the cathodic sputtering method (see Vol. II Abstract No. 151/III). Evaporating

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thin metal films on to plastics will reduce the resistivity of the surface to the order of a few ohms. Carbon applied to a surface either in the form of colloidal carbon suspended in an aqueous solution or deposited from gaseous hydrocarbons by pyrolytic methods will form a conducting film of a resistivity ranging from 1,000 Ω to several megohms or from 100 to 10,000 Ω respectively. Moisture collecting on a surface in the form of a film reduces surface resistivity effectively but these films are unstable and may evaporate under the action of the current passing through them. Finally, reference is made to the uses of films formed from wetting agents such as Lissapol N and Teepol 530. In particular the former material has a lasting effect on the reduction of static charges if applied to Perspex surfaces for instance.

Sommaire : On discute l'élimination des charges électro-statiques sur des surfaces isolantes en les recouvrant d'une couche conductrice.

Paper by J. S. Forrest *Brit. J. Appl. Phys.* Supp. 2, 1953 S.37-S.39

A Simplified Ionisation Gauge Circuit 92/I

See Abstract No. : 52/II

Improvements i.o.r.t. Metal Vaporisers 93/I

See Abstract No. : 61/III

14 — MECHANICAL SCIENCES — 14

Vacuum Tightness of Welded and Brazed Aluminium Containers 94/I

United Kingdom. In order to arrive at a proper assessment of the quality of welded and brazed aluminium joints with respect to vacuum tightness methodical tests were carried out on a number of containers of equal size (1.9 inch. diameter, 6 inch. long), fabricated from Alcoa 35 aluminium, each having a longitudinal butt-weld, two circular corner welds for the end plates and a fillet weld joining the exhaust tube to an end plate of the container. The brazed containers were of the same material and dimensions. The argon-shielded tungsten arc process was used for welding, and brazing was carried out at 1,120°F with the help of No. 714 brazing wire coated with a methanol paste of Alcoa No. 34 brazing flux. All details of the techniques employed are given. The containers were joined to the vacuum system by de Kothinsky cement. The vacuum system was served by a mercury diffusion pump and fitted with mercury cut-offs and a McLeod gauge. Aluminium, is known to form a sorbed film—largely water —on its surface or an oxide. According to calculations published by previous workers the containers tested in these experiments would adsorb almost 100 ml. STP of gas and water vapour, if exposed to the outdoor atmosphere for 10 months. Most of the film can be removed by heating *in vacuo* at 300°C but at higher temperatures the water will decompose and hydrogen gas will be evolved. The results of the investigation are given in tabular form and indicate that most of the gas evolved under the conditions of the test must have originated from the sorbed film on the metal, since the pressure was appreciably reduced when the container was cooled to -78°C in a dry ice-acetone bath eliminating the condensable gases. There is little difference in quality between a welded and a brazed container. In the case of the container W-4 and B-1 (see the tables partly reproduced below), the rate of evolution of permanent gas was found to be less than 2×10^{-5} ml. STP/hr.

Summary of Pressure Build-Up in Evacuated Welded Aluminium Containers

Container No.	Evacuation: Duration, Hr.	Evacuation: Temp., °C	Evacuation: Pressure, mm. Hg	Pressure Increase, mm. Hg: Test at +25°C: 1 Hr.	Test at +25°C: 10 Hr.	Test at +25°C: 100 Hr.	Test at -78°C: 10 Hr.	Test at -78°C: 100 Hr.
W-2	15 5	25 450	3×10^{-5} 2×10^{-5}	—	5×10^{-4}	26×10^{-4}	4×10^{-4}	21×10^{-4}
W-4	15	25 450	5×10^{-5} 4×10^{-5}	2.5×10^{-5}	1.1×10^{-4}	10×10^{-4}	1×10^{-4}	7×10^{-4}

Summary of Pressure Build-Up in Evacuated Brazed Aluminium Containers

Container No	Evacuation: Duration Hr.	Evacuation: Temp., °C	Evacuation: Pressure, mm. Hg	Pressure Increase mm. Hg: Test at +25°C: 1 Hr.	Test at +25°C: 50 Hr.	Test at +25°C: 100 Hr.	Test at -78°C: 1 Hr.	Test at -78°C: 50 Hr.	Test at -78°C: 100 Hr.
B-1	39 5	25 450	2×10^{-5} 2.5×10^{-5}	12×10^{-5}	10.5×10^{-5}	16×10^{-4}	—	10×10^{-4}	14×10^{-4}
B-2	19 5	25 450	22×10^{-5} 4×10^{-5}	6×10^{-4}	4×10^{-3}	—	3×10^{-4}	3×10^{-3}	—

Sommaire : Des récipients soudés et brazés en aluminium, établis avec différents types de joints ont été essayés à l'étanchéité du vide.

Article by M. A. Miller & A. S. Russell *Weld. J.* Feb. 1953 116-118

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Abstract No. and References

Plating as an Aid in the Brazing of Stainless Steel

95/I

United States. Plating of types 304 and 347 stainless steel with 1-2 mils Ni, and sintering of the plate in a H furnace at 1,000° for 30 minutes makes possible the use of simple H brazing techniques at low temperature and high dew point (25°) for obtaining vacuum-tight seals on intricate assemblies.

(*Chemical Abstracts*)

Sommaire : On décrit un procédé pour le brasage de l'acier inoxydable étanche au vide.

Article by A. Korbelak & E. C. Okress
Plating
39, 1952
1220-1222

Bursting Disc Assembly for Alternating Pressure and Vacuum at Elevated Temperatures

96/I

United Kingdom. Platens in presses for the moulding of thermosetting resins must be heated, preferably by steam, to a temperature of 350°F. In the particular case referred to a closed system had been proposed which consisted of a small gas-heated flash boiler raising the steam which condensed in passages in the body of the platen, the condensate returning to the boiler by gravity. The authorities waived the usual safety requirements such as the provision of a pressure gauge, safety valve, water level indicator and low water alarm but insisted on the provision of a thermometer and bursting disc assembly, working to the following specifications : The disc should burst at 300 lb./sq. inch steam pressure, withstand 200 lb./sq. inch, withstand the vacuum created by the condensation of the steam when the system is cool, be steam and airtight under pressure and vacuum and be easily replaceable. It was decided to mount the disc in a plug screwed into the side of the platen. Altogether three designs were developed. The first two showed deficiencies on test in the main due to differential expansion of the materials used and a danger of twisting during the tightening operation. The final design consisted of a plug polished at both ends, one end bears against the platen when the plug is screwed in, the other end serving as a seat for the disc. The disc of a free area of $\frac{1}{2}$ inch. diameter was held down by a hollow cone which in turn was secured by a clamping cap. The question of a suitable material for the disc proved more difficult and has only partly been solved so far. The stress calculations were based on equations by Lake and Inglis. As the disc diameter was small, the thickness had to be small. Nickel, otherwise well suited to withstand the continuously alternating stress, had to be ruled out for this reason. Materials of lower tensile strength were required. Aluminium and copper were useless due to work hardening and silver showed intolerable creep. An 0.5% gold-platinum alloy was finally chosen and preliminary tests with discs of 0.003 inch. thickness gave bursting pressures of 225 lb. sq. inch. and 275 lb. sq. inch for $\frac{3}{4}$ inch and $\frac{1}{2}$ inch diameters respectively. A thickness of 0.0033 inch proved to fulfill the specified requirements. The bursting of twenty discs, however, cut from two different strips, indicated that there was a difference in the bursting pressure which could not be entirely explained by variations in thickness. A closer metallurgical control of the material is therefore desirable and forms the subject of a separate investigation.

Sommaire : L'installation d'un disque dit ' eclatant ' est décrit, étanche au vide et alternativement à grande pression.

Paper by E. A. K. Patrick
Trans. I. Chem. E.
31, 2, 1953
114-119

15 — PHYSICS — 15

The Micro-Analysis of the Inert Gases

97/I

See Abstract No. : 111/I

The Motion of a Gas Cloud Expanding into a Vacuum

98/I

United States. The motion of interstellar clouds has been studied by considering the flow of a semi-infinite ideal gas cloud bordering on vacuum. These clouds consist almost exclusively of hydrogen and consequently the ratio of specific heats γ may be taken as 5/3, but the theory developed is valid for any value of γ. The effect of viscosity and heat conduction was neglected. The method of Riemann invariants and characteristic curves of the differential equations of the motion were used. These characteristic curves were then plotted to represent the flow in the case of problems previously examined by Burgers and Copson for a hydrogen cloud. Burgers' problem involved an initial discontinuity of density at the front of a homogeneous cloud while Copson stipulated an initial region of inhomogeneity between the vacuum surface and the main homogeneous cloud without any actual discontinuity in density. Both solutions involved gas flows which are continuous. The method of characteristic curves developed showed that unless special initial conditions are satisfied by the cloud the subsequent flow cannot remain indefinitely continuous and shock waves appear within the gas. Other initial conditions were also investigated and illustrated by characteristic flow diagrams. When the speed of sound increased linearly at first a limiting case of Copson's solution was obtained : The characteristics of one family shrank to a point on the wave front at the same instant of time. In the case of the flow remaining continuous the wave front was shown to move ultimately with a velocity of three times the speed of sound in the homogeneous cloud, whereas in the case of initial conditions leading to shock waves in the flow the wave-front attained even greater velocities. Thus the high eventual velocity of these gas clouds which was considered to be a physically unsatisfactory feature of previous solutions of the problem could not be avoided by a change in the initial inhomogeneities.

Sommaire : On a fait des recherches théoriques sur le mouvement d'un gaz d'hydrogène se répendant dans un vide interstellaire.

Article by D. C. Pack
Mon. Not. Roy. Astr. Soc.
113, Jan. 1953
43-51

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Abstract No. and References

Intensity Measurements in the Vacuum Ultra-Violet

99/I

United States. Recent progress in the research of the upper atmosphere has revived the interest in the intensity of the solar ultra-violet, but no complete spectral energy measurements are available in the region below 2,000 Å. Packer and Lock, the first to measure dispersed radiation in that range, used a thermocouple to obtain the special distribution of the H_2 spectrum at the exit slit of a vacuum monochromator, but the accuracy of their measurements suffered as a result of a gradual decrease in grating reflectance at the shorter wavelengths. The authors report on measurements carried out with a similar arrangement. Secondary detectors were used in the form of phosphor-coated photomultiplier tubes and windowless photoelectric cells with pure metal cathodes. The one-meter normal-incidence vacuum monochromator employed was designed to permit scanning from 300-4,500 Å at speeds of 300, 100, and 20 Å per minute. Three slits were available at each slit assembly, all 3mm. high but varying in width from 1mm. to 0.05 mm. the latter giving a resolution of about 1 Å. The pumping unit consisting of a 6-inch. oil diffusion pump and a mechanical fore pump giving an ultimate pressure of 1×10^{-5}mm. Hg. The light source was a water cooled d.c. discharge tube with a platinised quartz capillary of 4mm. diameter. The arc operated at 0.5 A. and 600 V. Normal cylinder gas was used for the experiments. The gas was admitted at a pressure of 0.5 mm. Hg. The thermocouple was mounted in one of the three slit positions of the exit slit turret. The secondary detector had a fixed position 3 cm. behind the exit slit. The second slit position was occupied by a 1 mm. slit and the third by an 0.05 mm. slit. During an early stage in the present investigation unexpectedly high spectral intensities were recorded, in particular, in the shorter wavelength region. This was found to be due to an accidental deposit of platinum which had evaporated from the capillary of the light source onto the grating. As a result of this, thermocouple measurements of the H_2 spectrum could be extended down to 850 Å. The intensity of the helium resonance line λ584 was measured with the thermocouple and found to be 0.025 micro-watt. With the help of an 1P21 tube coated with sodium salicylate the relative intensities of the first four members of the series (λ584, λ537, λ522, λ516) were found to be 1 : 0.025 : 0.004 : 0.0008. The values of the H_2 spectrum obtained by the authors are compared with the values obtained by Packer and Lock. Over a large part the curves differ by a factor of about two which can be explained either by the fact that the present workers used a larger thermocouple receiver area and a higher arc current, or by differences in the efficiency of the optical system. The article concludes with a brief discussion of the properties of sodium salicylate which make this material particularly suitable for use in a photomultiplier in connection with measurements of this kind.

Article by K. Watanabe & E.C.Y. Inn *J. Opt. Soc. Amer.* **43**, Jan. 1953 32-35

Sommaire : On décrit un système pour mesurer la distribution de l'énergie d'un spectre ultra-violet et les résultats obtenus.

Measurement of the Reflective Power of Thick Metallic Layers (Au, Pt, Cr) in the Schumann Range. Determination of Optical Constants

100/I

France. As far as is known to the author all information published on the reflectance of thick metallic layers in the Schumann range is confined to their performance at an angle of incidence of 18°. No data is available on the optical constants of metals in the vacuum ultra-violet. The author reports briefly on measurements carried out in the range from 1,100-2,000 Å at an angle of incidence of 18° and 45° from which the reflection factor and optical constants were derived by a method based on the electro-magnetic wave theory. Gold, platinum and chromium layers were used for the experiments, produced by the vacuum evaporation method. The results are given in graphs and in a table which is partly reproduced below. R_s^{45} is the reflection factor of

	Platinum		*Gold*		*Chromium*	
λ(Å).	$(R_s^{45})^2$.	R_p^{45}.	$(R_s^{45})^2$.	R_p^{45}.	$(R_s^{45})^2$.	R_p^{45}.
1,280	0.19	0.18	0.18	0.16	0.11	0.12
1,520	0.19	0.19	0.20	0.23	0.165	0.17
1,760	0.24	0.25	0.21	0.22	0.25	0.30
2,000	0.30	0.32	0.31	0.25	0.32	0.37

the S-component measured at an angle of 45°, and R_p^{45} is the reflection factor for the P-component measured at the same angle. Formulae are given for the refractive index and the absorption coefficient but it is not claimed that the latter gives accurate results. The fact that the values of the reflection for the two components are practically identical appears to permit the conclusion that the electro-magnetic theory applies in that range and can be used for the determination of the refractive index and the absorption coefficient.

Note by S. Robin *C.R. Acad. Sci., Paris* **236**, 16. 2. 1953 674-676

Sommaire : On a mesuré le coefficient de réflexion d'épaisses couches (évaporées sous vide) d'or, de platine, de chrome de l'ordre de 1,000 Å à 2,000 Å.

Apparatus for the Measurement of Reflecting Power in the Vacuum Ultra-Violet

101/I

France. This apparatus is used in conjunction with a monochromator for the vacuum ultra-violet. Measurements can be made of transmission and reflection coefficients for 18° and 45° incidence. Other special features

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include a reference beam for monitoring the incident intensity and arrangements to make measurements on evaporated layers produced *in situ*. Photomultipliers with sodium salicylate fluorescent screens are used as detectors.

(*Science Abstracts*)

Sommaire : On décrit un appareil servant à mesurer les coefficients de transmission et de réflexion de l'ultra-violet sous vide.

Letter by S. Robin & B. Vodar *J. Phys. Radium* 13, Oct. 1952 492

Infra-Red Spectra of Evaporated Films

See Abstract No. : 114/I

102/I

Mirror Coatings for Low Visible and High Infra-Red Reflectance

103/I

United States. Mirrors with selective reflectance properties such as low reflecting power in the visual range and high reflecting power in the infra-red range of the spectrum are of practical interest for use in infra-red equipment where reflectance in the visual range is undesirable. Copper and gold are known to have suitable optical properties but for mechanical reasons such as adherence and durability preference is given to aluminium and rhodium in the production of front surface mirrors. Both these materials have a high reflecting power in the visual range. This can be reduced by the application of a dielectric film effectively a quarter wavelength thick. In order to achieve zero reflectance at a particular wavelength the refractive index of the dielectric material would have to be of a higher order than is practicable. However, zero reflectance can be obtained by using an absorbent film. This can be demonstrated by evaporating TiO_2 from tungsten onto the mirror. During the evaporation process TiO_2 is slightly reduced and condenses as an absorbing material. As a result the reflectance of aluminium at $\lambda = 550$ mμ is decreased to less than 10%. This figure rises again to 60% on re-oxidising the film to non-absorbing TiO_2. A film of Si of the correct thickness and a film of Ge, if deposited in a poor vacuum (10^{-4}mm. Hg.) produce a similar effect on aluminium. Thus multilayer coatings from various absorbing materials can be designed which produce a minimum reflectance at more than one wavelength. Two such designs are discussed in detail. In the first Ge is deposited under carefully controlled conditions producing a reflectance minimum of 40% at $\lambda = 430$ mμ. On top of the Ge film a SiO film is deposited with the result that the reflectance at $\lambda = 430$ mμ becomes zero and a second point of zero reflectance is obtained at 600 mμ. The reflectance of an aluminium mirror treated in this manner is 2% in the visual range, rises rapidly at 0.8 μ to reach 80% at 1 μ, 90% at 1.2 μ and is equal to uncoated aluminium in the range beyond 1.5 μ. The occurrence of the double minimum is explained by means of a vector diagram. In the second design the Ge film is replaced by a film unit, SiO+semi-transparent aluminium, calling for a total of 3 films to be deposited on the aluminium mirror in the order : SiO-Al-SiO filter-mirror. The first SiO layer decreases the reflection of the mirror to 60%. Then aluminium is deposited until the reflectance at the controlled wavelength, after passing through zero, reaches a value of 15–20%. The top SiO film reduces the reflectance again to zero.

Sommaire : On décrit les moyens d'obtenir par évaporation sous vide des miroirs d'aluminium recouverts d'une couche absorbante de façon à obtenir une faible réflexion dans le spectre visible et une grande réflexion dans l'infra-rouge.

Paper by G. Hass, H. H. Schroeder & A. F. Turner *Conference Opt. Soc. Amer.* March 1953

The Limit of Accuracy in the Measurement of the Thickness of a Thin Film Using the Koester Interference Double Prism

104/I

Germany. An arrangement permitting the application of photometric methods is described. Fringe width is narrowed by multi-photographic copying and this permits an accurate measurement of fringe separation. Three photometric methods of evaluating displacements are described. The method permits the measurement of the thickness of deposited thin films. A square centrimetre film can be measured with the thickness down to about 20 mμ.

(*Science Abstracts*)

Sommaire : On décrit une méthode photométrique servant à mesurer l'épaisseur des couches minces.

Article by U. Zorll *Optik* 9, No. 10. 1952 449-455

Shadow Microscopy for Measurement of Height

105/I

United Kingdom. A shadow technique for use in light microscope work is described which in a manner analogous to the metallic shadow casting technique used in electron microscope work facilitates the determination of the height of small objects and the assessment of surface characteristics. The object is placed on a slide with a flat fine-grained scattering surface to ensure a clear demarcation of the shadow. In order to obtain the right surface properties it is proposed to deposit a silver film on the slide and to expose the layer to vapours of aqua regia, which will produce the desired finish. In position the object on the slide is illuminated by a parallel beam of light of 1 mm. diameter passing through a lens and stopped down by an iris. If the light beam is directed to the object at a grazing angle of slightly less than 3° the length of the shadow thrown will be about 20 × the object height. Assuming a microscope magnification of 500 × a total magnification of 10,000 can be obtained. Balls of Lycopodium powder which can be easily measured should be placed on the slide for calibration purposes. The ratio of the length to the width of the shadow thrown by the ball gives the magnification ratio. In some applications, it will be advantageous to create the required matt surface *in situ*, for instance, where the features of a crystal are to be examined. Illustrations are shown of typical applications such as the examination of spores of *Trichoderma Viride*.

Sommaire : On décrit une technique d'ombre pour le microscope d'optique permettant de mesurer la dimension de petits objets analogue a celle employée pour le microscope électronique.

Letter by S. Tolansky *Nature* 171, 28.3.53 564-566

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Preparation of a Tritiated Self-Luminescent Material 106/I

See Abstract No. : 112/I

Thermal Conductivity of Glass Wool at Low Temperatures under Low Pressure of the Air 107/I

France. The heat conductibility of glass wool, placed between the double walls of a vessel filled with liquid air, is studied as a function of the thickness of the fibres and the residual pressure of the included air. The evaporation of the liquid air obtained with the glass wool is compared to that obtained with vacuum insulation the wall being silvered or not. The curves permit to determine the optimal density of the glass wool filling.
(*Bull. Inst. Int. du Froid*)

Paper by O. van Paemel *C. R. Comm. 1 & 2 I. I. F., Louvain* June 1952

Sommaire : On a étudié la conductibilité thermique de la laine de verre par rapport au problème de l'isolation sous vide.

16 — CHEMISTRY — 16

Dehydrated Meat 108/I

See Abstract No. : 30/IV

Studies on the Stability of Lyophilised BCG Vaccine 109/I

See Abstract No. : 29/IV

Industrial Applications of Activated Alumina 110/I

See Abstract No : 64/II

The Micro-Analysis of the Inert Gases 111/I

United Kingdom. The author surveys the existing methods of analysing very small quantities of noble gases and gives a detailed account of the equipment and procedures employed at the Londonderry Laboratory for Radio-Chemistry at Durham. In the introduction reference is made to Ramsay's work on noble gases and Ramsay's and Soddy's investigation in 1903 proving for the first time the production of helium gas by radio-active substances. Collie and Paterson, members of Ramsay's laboratory, believed to have found evidence that electric discharges can produce helium and neon which appeared to confirm Ramsay's thesis that radio-active radiation can transmute ordinary stable elements. After the first World War, when vacuum techniques had sufficiently progressed the author could disprove Collie and Paterson's assumptions by demonstrating that a similar amount of helium collected in an electric bulb burning in open air for a few days but none if the bulb was operated under water, *i.e.*, diffusion through the hot glass accounted for the helium detected in the vessel at the time. A detailed description of the Durham apparatus is given and illustrations. Attention is drawn to certain special devices incorporated in the equipment, *e.g.* the palladium furnace for burning last traces of hydrogen, the barium furnace for argon, krypton and xenon analysis, a fractionating column for the separation of helium and neon, Pirani gauges for the measurement of these gases and vessels for air-free dissolution. Details of recent work carried out in Durham are given. In order to prove the reality of artificial transmutation of elements by chemical methods boron was bombarded with neutrons from a radium-beryllium source yielding helium, for the first time artificially produced in measurable quantities in this way. Another important field is that of geological age determinations. Normally the mineral under investigation is heated to set free the helium held by the material but in the case of iron meteorites this method is unsatisfactory and the helium is released by dissolving the iron chemically. The helium so collected can be measured directly in a Pirani gauge. A method is described which permits the determination of uranium and thorium in quantities as low as 10^{-8} g. per gram of meteorites. Application of the most refined methods for the determination of helium to 50 samples of iron meteorites indicated that all meteorites were 2 or 3 times as old as terrestrial rock. It was suspected that the amount of helium determined in these investigations was not entirely of radio-active origin but partly produced by cosmic rays to which the meteorites were exposed during their flight. This was proved when the helium content of five iron meteorites was examined for its isotopic composition, and was found to contain up to 24% of helium 3. Normally helium 3 is present in radio-active minerals in quantities up to 10^{-5}% and in atmospheric helium in quantities up to 10^{-4}%. These findings may help to improve existing knowledge on the origin of the solar system. Finally, reference is made to research of the upper atmosphere. Usually, the composition of the air at great heights is assessed by determining its oxygen content. But this method is not entirely satisfactory because the oxygen may have reacted with the material of the collecting vessels. Instead it was suggested to

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test the ratio of the noble gases to nitrogen postulating that if gravitational separation took place the ratio of helium and neon to nitrogen would be increased and the ratio of argon to nitrogen decreased. This consideration was proved to be correct on samples collected over New Mexico at heights beyond 60 kilometres. It should be noted that the collecting vessels have a capacity of 8 litres but no more than one c.c. of air was collected by rockets at heights of 65 kilometres and at 100 kilometres the amount collected is not expected to exceed 1/10th of the c.c.

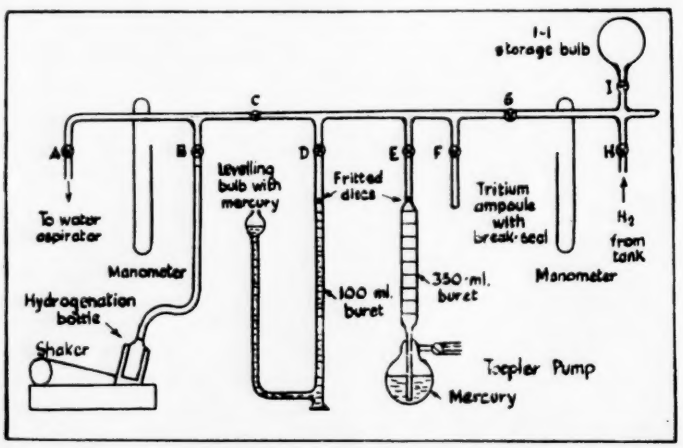
Sommaire : Un. compte-rendu général des méthodes actuelles et des instruments employés dans la micro-analyse des gaz rares au département de Radio Chemistry de l'Université de Durham.

Article by F. A. Paneth *Endeavour* 12, Jan. 1953 5/17

Preparation of a Tritiated Self-Luminescent Material

112/I

United States. As many organic aromatic crystalline materials are known to fluoresce if exposed to ionising radiation it was expected that a self luminescent material could be prepared by introducing a radio-isotope into the molecule of one of these compounds. The authors report on a method of preparing a self-luminescent material by introducing tritium. This isotope was chosen because, (1) tritium-activated materials can be handled with comparative safety, (2) tritiated phosphors should yield a fairly constant luminosity and (3) tritium of comparatively high specific activity is available at low cost. The self-luminescent material was prepared by reducing diphenylacetylene with tritium to *cis*-stilbene and the subsequent isomerisation of *cis*-stilbene by heat to *trans*-stilbene. The tritiation was carried out in an apparatus illustrated in a sketch reproduced below which is of general use for the tritiation of organic compounds containing multiple bonds.



The hydrogeneration bottle of a volume of 250 ml. was filled with a solution of 1 g. diphenylacetylene in 7 ml. of dry benzene and 0.05 g. of 1% palladium on calcium carbonate catalyst. The bottle was placed in a mechanical shaker. Subsequently the entire system was evacuated with the help of the water aspirator shown on the left. Then the hydrogen flask on the extreme right was filled and subsequently the whole system flushed four times with the hydrogen from the storage bulb. This was followed by a process of evacuation at the end of which the stopcocks B, C and D were closed. Then tritium was transferred from the ampoule to the 350 ml. buret by reducing the mercury level in the buret with the help of a Toepler pump at the bottom of the buret. The stopcock E was closed temporarily and hydrogen admitted to the tritium ampoule, until a pressure of 1 atmosphere was reached. The stopcock E was reopened and by repeated pumping with the Toepler pump a thorough mixture of tritium and protium was obtained. When 100 ml. of gas at 1 atmosphere had collected in the buret the gas was transferred to the hydrogenation bottle. After the transfer the pressure in the hydrogenation bottle was brought up to 1 atmosphere by admitting hydrogen from the storage bulb and the stopcocks B, D and E closed. The stopcock D was opened and hydrogen transferred to the 100 ml. buret until a pressure of 1 atmosphere had been reached as indicated by the levelling bulb. At this stage the shaker was started and the volume of gas absorbed in the reduction process measured by means of the 100 ml. buret. After the theoretical amount of hydrogen had reacted, the reaction was stopped and the apparatus flushed with air. The liquid *cis*-stilbene obtained from the reaction was pipetted into a tube, frozen in liquid nitrogen, degassed, refrozen and sealed under vacuum. Subsequently the tube was heated in a furnace for 90 mins. at 315°C, yielding *trans*-stilbene in crystalline form. The final product was purified before use. The light output from tritiated stilbene is shown in a graph as a function of the mass of the material and compared with that of a radium-activated self-luminescent ZnS (Ag) phosphor screen. The apparent brightness of *trans*-stilbene is about 1 microlambert for a sample of 30 mg./sq. cm. thickness. The conversion efficiency of light production is approximately 0.6%.

Sommaire : Une substance phosphorescente a été preparée en réduisant diphenylacetylene par tritium en cis-stilbene et trans-stilbene de ce dernier par la chaleur.

Article by I. A. Bernstein, W. Bennett, M. Fields & E. C. Farmer *Nucleonics* 11, Feb. 1953 64-66

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Abstract No. and References

Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy 113/I

See Abstract No. : 54/II

Infra-Red Spectra of Evaporated Films 114/I

United States. It has not been widely recognised yet that the vacuum evaporation technique can be used to prepare thin films of organic compounds which can serve as specimens for absorption spectroscopy. The condensed film may be identical to the original compound or represent an isomeric or polymorphic form of it. Alternatively, a new compound may have been formed as a result of decomposition. To illustrate the first case the infra-red spectra of mineral oil and hexachlorobutadiene mulls and of an evaporated film of anthracene are shown. The spectra are the same but the evaporated specimen shows higher backgrouhd transmittance and stronger and sharper absorption bands. The x-ray powder pattern of the evaporated material shows less detail. To illustrate the second case the infra-red spectra of a mull of benzidine yellow made from o-acetoacetotoluidide and of the same material evaporated are shown. New bands appeared in the latter at 4.42 and 12.75 micron indicating a change. However, after heating the evaporated sample at 165°C for about 100 hours the material returned to its original state confirming that no decomposition had occurred. It was not decided in that case whether the new material was an isomer or a polymorph. No compound has been found yet to illustrate the third case. The method described offers the following advantages : The elimination of the need for two mulling media, increased background transmittance, greater sharpness of absorption and good control of specimen thickness. But it cannot be used for completely unknown materials. The experiments were carried out in an evaporator with a bell jar of 5½ inch. diameter and 8 inch. height. The operational pressure of 10^{-4}mm. Hg was produced by an oil diffusion pump. Some data on the electrical equipment is given.

Article by J. E. Tyler & S. A. Ehrhardt *Analyt. Chem.* **25**, March 1953 390-394

Sommaire : On discute brèvement l'utilisation des procédés de déposition sous vide pour la préparation des spécimens de composés organiques dans la spectroscopie à absorption.

Thermal Degradation of Polystyrene 115/I

United States. Grassie and Melville published a method of investigating the degradation of a polymer by determining its volatile degradation products. The material (polymethylmethacrylate) was placed into a continuously pumped system and heated. A constriction in the system limited the pumping speed to a constant value. Thus, the measured pressure was directly proportional to the rate of production of monomer. The author investigated the degradation of thermally polymerised styrene by the same method using a mass spectrometer as pressure gauge. 25 mg. of the material were deposited in the form of a film, approximately 10^{-4} cm. thick, on a quartz tube with an area of 150 cm². The quartz tube was directly sealed to the sample inlet tube of the mass spectrometer, the latter tube constituting the constriction required for the limitation of the pumping speed. All measurements were taken at a maximum pressure of 2×10^{-7}mm. Hg in the spectrometer tube. According to the calculations the corresponding pressure of the monomer in the quartz tube was about 2×10^{-4}mm. Hg. Initial outgassing, 4 hours at 80°C followed by 12 hours at 30°C, indicated benzene as the predominant volatile present. The temperature was then raised slowly to reach 110°C after 5 hours. At this stage the volatiles indicated were benzene, methylcyclohexane, toluene, ethyl benzene and acetic acid. The latter was the predominant volatile when 145°C had been reached after a further 9 hours. For 16 hours at this temperature the evolution of volatiles dropped and the temperature could be raised slowly to 220°C. Now for for the first time, small amounts of styrene could be identified among the volatiles. At the peak temperature of 310° proper decomposition into styrene, benzene and toluene was noticed but gas evolution was of short duration. The temperature was then reduced to 230° and the material kept at that temperature for about 70 hours, when the evolution of gas ceased altogether. Up to this point no more than 1% of the sample had been volatilised. The persistent indication of the solvent (benzene), was rather unexpected and the presence of acetic acid and methylcyclohexane could not be explained. Finally the sample was gradually heated up to 330° over a period of ten days. Proper decomposition was believed to have taken place during that treatment. The amounts of styrene, benzene and toluene liberated proved to depend only on the temperature. A graph is shown of the principal peak heights for styrene, benzene and toluene plotted against the reciprocal of the temperature, from which the activation energies for the production of the materials could be determined. Benzene and toluene had the same activation energies but styrene had a higher activation energy. The rate of evolution of styrene was calculated to be 7×10^{-3}% of the sample per minute at 330°C. Reference is made to Madorsky's experiments which indicated a higher rate of decomposition but this may be due to differences in the experimental procedures.

Letter by P. D. Zemany *Nature* **171**, 28.2.53 391-392

Sommaire : On a déterminé par un procédé sous vide la décomposition en fonction de la temperature du styrene polymerisé thermiquement.

Alkali-Refined Linseed Oil 116/I

United Kingdom. When refining linseed oil for use in the paint and varnish industries it is necessary to remove all impurities. The crude linseed oil contains fatty acids, phospharides and mucilaginous matter. In order to make the oil suitable for use with paints and varnishes all foreign matter must be eliminated by a refining process, normally a treatment with alkali (caustic soda) which neutralises the free fatty acid, and hydrolyses and precipitates the ' break ' material. The success of the purification process depends on the efficiency with which the precipitated material is separated. A continuous and automatically controlled process is described

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which is based on a method developed in the U.S.A. In this process the heating time and the time of contact between oil and alkali are reduced to a minimum. Provision is made to add varying amounts of alkali to suit any grade of crude oil, giving a refined oil of constant characteristics. After the crude oil has been heated to 70–80°F. in a tank fitted with an agitator to prevent settlement of the impurities, it is pumped to a high speed mixer where it is mixed with a pre-determined amount of caustic soda solution by a proportionometer. The resultant emulsion passes through a continuous heater, where the temperature is raised to 130–160°F, causing the emulsion to break. It is then fed to a centrifuge where the two components are separated and discharged into separate tanks. The neutralised oil is transferred to another tank, heated to 160°F, hot water of 180°F equivalent to 10% of the oil weight is added, and the mixture admitted to a water washing centrifuge in measured quantities. Each individual wash takes about 25 minutes. In order to make certain that all traces of soap are removed a second wash is provided. After the second wash the oil has a moisture content of 0.5%. This is reduced to 0.05% in a continuous vacuum dryer by a flash evaporation process. The dryer consists of a vertically mounted cylindrical chamber, evacuated to a pressure of between 1 and 2 inch. Hg by a steam ejector. Baffles at the top of the chamber prevent entrainment of oil in the vapour of the exhaust system. A mechanical vacuum pump is available should the steam fail, or a higher vacuum be needed. The oil is charged to the dryer by the action of the vacuum through groups of nozzles at different heights. Each group of nozzles is controlled by a solenoid valve, which in turn is controlled by a float in the feed tank, to keep the chamber running at full capacity. Safety devices prevent overcharging. After drying the oil is pumped away for storage, or to a vacuum earth bleaching plant, depending on requirements.

Article by E. J. Kelly *Paint, Oil Colour J.* **121**, 20.6.52 1421-1422

Sommaire : Rapport detaillé sur les méthodes de raffinage de l'huile de lin, utilisant le vide, employées dans l'industrie des vernis et peintures.

Distillation Apparatus for Refining Fats and Oils — 117/I

See Abstract No. : 88/III

Improvements in the Functioning of Continuous Vacuum Distillation or Rectification Apparatus — 118/I

See Abstract No. : 86/III

Falling Film Type of Vacuum Still with Rotating Partial Condenser — 119/I

See Abstract No. : 87/III

17 — METALLURGY — 17

Casting Under Extreme Conditions — 120/I

See Abstract No. : 80/III

Electron Diffraction Investigation of Polished Surfaces — 121/I

United States. The electron diffraction (reflection) patterns of polished surfaces of metals show a pair of very diffused rings or halos with sin θ/η values almost identical for all materials. These so called ' polish ' patterns are interpreted to indicate that the polish layer is amorphous. The author examined the polished surfaces of silicon, germanium, boron and beryllium by electron diffraction. All four materials showed the same typical pattern. On the other hand, it has been established that many polished silicon and germanium surfaces contain fairly large crystallites of the base material and of the abrasives used. It is known from x-ray diffraction studies of liquid metals that the pattern obtained depends on the atomic radius. The atomic radius of polished boron can be expected to be much smaller than the radii of other metals. It is unlikely, therefore, that the electron diffraction pattern of boron and beryllium, for instance, could be the same. Further, various measurements showed that silicon surfaces which had been polished were different in structure from those which had been polished and then heated in air at 1,100°C but the electron diffraction pattern obtained was still the same. In view of this evidence it is believed that the typical ' polish ' pattern obtained in electron diffraction examinations is originated by the film of a substance which is readily adsorbed on the metal surface such as hydro carbon films which it is difficult to remove from some polished surfaces. The 'polish' pattern has great similarity to those obtained from several oils. This new interpretation does not exclude the possibility that in certain cases the polished surface may be amorphous.

Letter by T. S. Renzema *J. Appl. Phys.* **23**, Dec. 1952 1412

Sommaire : En vue de certaines expériences on suggère que le ' polish pattern ' obtenu par la diffraction des electrons sur des surfaces de metal poli provient de couches d'hydrocarbones adsorbées.

Sorbed Gases and High Vacuum Systems — 122/I

See Abstract No. : 79/I

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Improvements i.o.r.t. the Production of Coatings on Ferrous Articles 123/I

United Kingdom. An improved method is described for producing metal coatings on ferrous articles by diffusion. In the past the article to be treated was placed in a furnace and embedded in ceramic chippings which had been previously impregnated with a chromium halide. On heating the chromium halide vapour reacted with the iron at the surface of the article as a result of which the chromium coating was formed. This procedure had the disadvantage that the chippings tended to adhere to the article. In the new method, the article and the impregnated material are kept separate in the furnace. In preparation of the coating process blocks of porcelain or sillimanite are impregnated by heating them to a temperature between 700°C and 1,400°C in the presence of an ammonium chloride-chromium mixture until maximum absorption is reached. The impregnated blocks are stored in a warm dry atmosphere until required. For the coating treatment the blocks are moved into the furance, lining the walls, and the articles are placed on a shelf in the centre. Subsequently the furnace is evacuated to a pressure between 0.5–40mm. Hg and heating commenced. The treatment is discontinued when the desired coating thickness has been obtained. The blocks may be used repeatedly until the coating of a test specimen falls below the required standard. They are then re-impregnated in the manner described.

P. H. Lawrence, R. Rainsforth, A. Braybrook & The Birmingham Small Arms Co. Ltd. *Brit. Pat.* 686,097

Sommaire : Le brèvet décrit une méthode de production d'un revêtement métallique sur des articles en métal fereux par méthode pyrolytique.

Behaviour of Refractory Oxides in Contact with Metals at High Temperatures 124/I

See Abstract No. : 67/II

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The Solubility of Oxygen in Gamma Iron 125/I

United Kingdom. The ' solubility of oxygen in iron ' is defined as the maximum amount of oxygen that can be taken up by iron before a separate oxide phase is formed. Various mechanical properties, the corrosion behaviour and the structure of iron may be influenced by oxygen present in this form. It was the object of the investigation reported here to establish whether the amount of oxygen which can be dissolved in solid iron can be measured. Three different experimental methods have been applied. All were based on the same principle : Thin foil of extremely pure iron was brought to equilibrium with an H_2–H_2O or CO–CO_2 gas mixture, the partial pressure of the oxygen being kept slightly smaller than that facilitating the formation of FeO. The amount of oxygen taken up by the iron was measured and from a knowledge of the Fe-FeO equilibrium it was possible to calculate how much oxygen would have been absorbed by iron in equilibrium with FeO assuming Sievert's law to hold for oxygen in solid iron as it does for liquid iron. The first two methods did not yield acceptable results. The arrangement used for the final method is shown in a sketch. A Pyrex glass system was used, served by a mercury diffusion pump. The iron foil, 5 inch. × 0.5 inch. × approximately 0.003 inch. was placed in a reaction vessel and heated electrically *via* copper leads. Provision was made for easy replacement of the strip. After the admission of purified dry hydrogen at a pressure of 50 mm. Hg the strip was heated to 1,250°C and maintained at that temperature for 10 minutes. The current was switched off, the gas withdrawn and the oxygen removed was determined by measuring the amount of water which had formed in the reaction vessel. This procedure was repeated several times to ensure complete deoxidation. Then, the CO–CO_2 mixture was admitted at 100 mm. Hg for 10 minutes and the strip heated to the required temperature. Finally, hydrogen was admitted and the same procedure employed for the determination of the oxygen taken up by the iron from

Table III. Summary of Final Results with High-Purity Irons

(*Oxygen absorbed from mixture with* CO_2-CO = 0.171)

Iron Used	*Temperature* (°C ± 20°)	*Gas*	*Wt. per cent. Oxygen absorbed* (±0.002)
A	1325	S	0.004*
	1365	S	0.004
	1412	S	0.004
N	1330	S	0.002
	1423	S	0.002
U	1330	S	0.002*
	1350	Str.	0.002
	1423	Str.	0.002
Z	1330	S	0.002
	1412	S	0.002

S = static gas mixture at 100 mm. pressure.
Str. = streaming at mean linear flow rate of 1 cm./sec.
* = Mean of two determinations.

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Table IV. Summary of Results with Iron-Maganese Alloys

Alloy	*Temperature* (°C ± 20°)	CO_2/CO *ratio*	*Per cent. oxygen absorbed* (±0.005)
X (2.89% Mn)	1330	0.171	0.1
Y (0.18% Mn)	1330	0.114	0.034
"	1412	0.114	0.030
"	1330	0.171	0.050
"	1412	0.171	0.041

(CO-CO_2 mixture static at 100 mm. pressure).

the CO–CO_2 mixture as in the case of the initial deoxidation. The CO–CO_2 mixtures used contained up to 14.6%CO_2 (by volume) giving a ratio of CO_2/CO corresponding to about two thirds of the critical ratio required to form FeO in the range of 1,200 to 1,500°C. The iron samples were supplied from several sources of which A, N and U were high purity iron and X, Y and Z carried alloyed materials as follows : X 2.89% Mn., Y 0.18% Mn and Z 0.013% Si. The strips had to be replaced after about ten runs. The results of the experiments, estimated to be accurate within 0.002% are shown in Tables 3 and 4 reproduced above. The authors believe that the lowest values are correct. In a detailed discussion the reasons are given which led to this conclusion. Assuming that the figure of 0.002% shown in Table 3 is typical for the behaviour of high purity iron in the γ range and applying Sievert's law for a mean temperature in the middle of that range, the theoretical limit of the solubility of iron in equilibrium with FeO can be estimated to be 0.003 ±0.003% by weight. But, since all samples whatever their purity contained impurities in excess of 0.003% it is possible that the true solubility is less than 0.001%, *i.e.* experimentally immeasurable and that the experiments give ' apparent ' rather than true solubility. But the experiments have proved that slightly impure iron can absorb oxygen quantities of practical significance before the phase is reached where an oxide of iron is formed. This is attributed to ' internal oxidation ' resulting in ' inclusions ' of oxides of elements with a higher affinity for oxygen than iron itself. The structural reason for the very low solubility of oxygen in iron is almost certainly a steric one. O ions believed to form if oxygen is in contact with iron are too large to enter the lattice of γ iron interstitially or substitutionally. The only alternative mechanism is intergranular diffusion.

Sommaire : Un rapport concernant la solubilité de l'oxygène dans le fer.

Article by J. A. Kitchener, J. O'M. Bockris Molly Gleiser & J. W. Evans *Acta Metallurg* 1, Jan. 1953 93-101

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Forging of Arc-Melted Chromium 126/I

See Abstract No. : 84/III

Titanium : A Survey 127/I

See Abstract No. : 81 III

Estimation of the Quality of Zirconium Powder Used in the Production of Vacuum Tubes 128/I

See Abstract No. : 76 I

A Technique for Making Molybdenum Crucibles for Ceramic Research 129/I

United States. In the course of an investigation of viscosities at high temperatures cylindrical crucibles were required of a height considerably greater than their diameter and the shape of which had to be changeable. A simple method of fabricating the crucibles was developed. The cylindrical form was made from rayon cloth held under spring-actuated longitudinal tension on a support which consisted of a brass rod fitted with a brass cylinder of the required crucible diameter at either end. One cylinder was sliding on the rod and could be fixed in any position governed by the length of the crucible to be fabricated. The cloth was tied to the end of each of the cylinders and coated with fast-drying model aeroplane dope. Then several layers of molybdenum powder suspended in a starch solution were brushed on allowing sufficient time for each coat to dry. Subsequently the cloth was removed from the holder, dope thinner applied to the inside, the cloth removed from the coating (now rigid) and the ends of the cylinder were ground with emery paper to suit. Where the crucible had to be fitted with a bottom molybdenum was built up on a small piece of unglazed tile coated with a layer of dope. Whilst the molybdenum build-up was still wet the cylinder was joined to it, and the assembly allowed to dry over night. After immersion in a bath of thinner for about 15 minutes the tile could be removed from the assembly. Finally, the crucible was fired in an induction furnace at about 2,000°C in hydrogen. A linear shrinkage

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of about 20% occurred during this process. Polishing the outside of the finished crucible with a silicon carbide stopped slight porosity noticed after firing. The crucible thereafter held liquids without leakage. Crucibles fabricated by this method have been employed for vacuum melting aluminium oxide and mixtures of aluminium oxide and calcium oxide.

Sommaire : Description d'un procédé relativement simple pour la fabrication de creusets en molybdène.

Report by H. T. Smyth & R. H. Meinken *N. J. Cer. Res. Sta. Tech. Rep* No. 3 June 1952 5 pp.

New Dispenser-Type Thermionic Cathode 130/I

See Abstract No. : 36/IV

19 — SCIENCES (other than the above) — 19

Methods of Increasing the Electrical Conductivity of Surfaces 131/I

See Abstract No. : 91/I

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II — Vacuum Apparatus and Auxiliaries — II

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20 — PUMPS — 20

Development of a Large, Linear Jet, Mercury Diffusion Pump Having High Pumping Speeds in the 10^{-6}mm. Mercury Absolute Pressure Range — 41/II

United States. At the present stage large banks of conventional diffusion pumps are required, where extraordinarily large vessels have to be pumped down to a high vacuum rapidly. This involves great expense for pumps, piping, controls and instrumentation. It appeared justifiable, therefore, to review present pump designs and to try a different approach. This report gives information on the design and performance of an experimental mercury vapour pump with a rectangular aperture of 72 inch. × 28 inch. and fitted with two linear jets, one backing the other. The mercury vapour was supplied from an external boiler and the pump was cooled by liquid refrigerant passing through copper tubes. The principle of the pump was based on a pump design conceived and tested by K. M. Simpson previously. The jets could be moved down the pump body. As a result the aperture into which they worked was changed. Other variations tried on one or both nozzles were: throat width, angle of nozzle 'skirt', length of the 'skirt' and wattage input. Super-heating the mercury vapour and copper plating the walls had little effect on the pump performance. The Ho coefficient of the pump increased with increasing aperture without reaching a maximum, proving that a pump 28 inch. wide with a 22 inch. total pump throat is a practical proposition. The pump reached a maximum speed of 50,000 l./sec. at 5×10^{-7}mm. Hg at an input coolant temperature of −14°C. At a coolant temperature of 5°C the speed fell to 35,700 l./sec. In the latter case the Ho coefficient was 0.405. An 8 foot linear pump of the new design takes about the same floor area as two 32 inch. diffusion pumps of a well-known make. The table reproduced below gives a comparison of the capacities available. Other advantages are that the pump can be

Description	*28 inch. × 8 ft. Linear Pump*	*Two 32 inch. Pumps*
Pumping speed using 5°C (41°F) cooling temperature	47,600 l./sec.	24,000 l./sec.
Pumping speed using 15°C (59°F) cooling temperature	32,000 l./sec.	16,000 l./sec.
Pressure at maximum pumping speed	1×10^{-6}mm. Hg	1×10^{-4}mm. Hg
Ultimate vacuum at base pressure	5×10^{-8}mm. Hg	1×10^{-7}mm. Hg
Power required	6.7 kW	12 kW
Fore pressure tolerance at above conditions	30 micron	150 micron

made extremely long, the boiler can be external to any radioactive shielding that may be required, and a single large boiler may supply many pumps. With valves in the vapour lines, a stop valve in the mercury return line and a small valve in the linear pump fore line it would be possible to rough the system through the mercury pump. This eliminates large heavy valves on the high vacuum side of the linear pump. The conclusions arrived at were that large pumps of similar design were practical for vacuum systems requiring large pumping capacity at pressures less than 10^{-6}mm. Hg.

Sommaire: Rapport sur la construction et la performance d'un montage expérimental d'une grosse pompe à diffusion de mercure et jet linéaire.

Report by E. R. Lind J. F. Steinhaus & California Research & Development Co. *A.E.C. Report* No. MTA-14 March 1953

Improvements i.o.r.t. High-Vacuum Oil Diffusion Pumps — 42/II

United States. A method is described of winding cooling coils on booster diffusion pumps employing vertically disposed umbrella-type jets. The cooling is applied first to the region of the upper jets and to the fore line and then round the body near the boiler where the water is warmed before passing back to the wall surrounding the fore jet. The object is to ensure that condensed oil returning to the boiler is warm enough to pass in a thin film through the small gap between the fore jet and the pump wall, rather than in a thick viscous layer which would tend to block the gap and impede pumping. A valve is included in the coil to control the rate of flow. Figures are quoted giving typical dimensions and temperatures.

Sommaire: Les serpentines de refroidissement d'une pompe à éjecteur sont placés dans une position assurant que l'huile condensée retournant à la chaudière forme une couche mince, évitant de bloquer des passages étroits de la pompe.

R. B. Lawrance & National Research Corporation *Brit. Pat.* 686,659

The Cooling and Baffling of Diffusion Pumps — 43/II

United Kingdom. Some observations are reported on the relationship between pump body temperature and performance of a diffusion pump. This leads to an examination of suitable cooling coil arrangements and various experimental baffle designs.

Sommaire: Recherches sur les relations entre la température du corps d'une pompe à diffusion et ses performances.

Letter by L. Riddiford *Vacuum* 3, Jan. 1953 49-51

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Ejectors Show Low First Cost

44/II

United States. The initial and running cost of steam ejectors are surveyed There are four different general types of ejectors : Single stage; multi-stage, non condensing; multi-stage, condensing; multi-stage, combining condensing and non-condensing stages. A table reproduced below gives the suction pressures attainable in multi-stage ejectors. The ejector capacity is expressed in lb. of dry air removed per hour. For reasons of economy, the condensable portion of the pumped gas mixture is removed in a pre-condenser and/or the inter-condenser unit of the ejector system. The capacity can only be altered by changing the suction pressure or the ejector dimensions. Where a flexible system is required, several ejector systems working parallel to each other can be provided, shutting down some of the parallel elements as desired. The cost is influenced by the following factors : Operating steam pressure, normally standardised at 100 psig. where the ejector discharges into the atmosphere; back pressure, increased only where use can be made of discharged low-pressure steam; number of nozzles supplying the steam (preferably one large nozzle only to avoid clogging); capacity; suction pressure; number of stages, increasing the initial cost but reducing the steam and water consumption; the condenser design, barometric or shell-and-tube type, and finally the materials of construction. Cost graphs are given for 2-stage, 3-stage and 4-stage ejector systems showing the initial cost and the running cost in terms of steam and water consumption for various steam pressures in each case. The expenses for steam and water per annum are normally higher than the installed cost. Maintenance is negligible. It is stated by the author that ejectors are often preferred to mechanical pumps in industrial operations due to the simplicity of the design (no moving parts), the absence of any lubricating material and the low maintenance cost.

Suction Pressures Attainable in Multi-Stage Jets

No. of Stages in Series	*Suction Pressure Range mm. Hg Abs.*
1	760 — 75
2	125 — 5
3	50 — 3
4	5 — 0.2
5	0.3 — 0.05

Sommaire : Etude des frais initiaux et d'opération d'éjecteurs à vapeur.

Article by J. C. Tallman *Chem. Engng.* January 1953 176-179

A Simple Air Ejector as a Suction Motor for Dry-Boxes

45/II

United Kingdom. Details are given of the construction of an air ejector for pumping dry-boxes. The advantages of an ejector over the normally used fan-type blower are: Higher reliability and the possibility of diluting the radio-active waste with a large amount of non-active material. In order to obtain a suitable design, giving maximum rate of flow for minimum pressure (of compressed air) applied, methodic experiments were carried out with an adjustable metal ejector built from interchangeable standard components of various sizes. Jets of 0.2, 0.3, 0.4, 0.5 and 0.7 cm. internal diameter and venturis of 0.6, 1.0, 1.2 and 1.6 cm. internal diameter were used. The distance between jet and venturi could be varied from 1-6 cm. The dry-box used in the experiments measured 5 ft. 6 inch. × 2 ft. 6 inch. × 2 ft. 6 inch. The duct had a diameter of 2½ inch. The pressure of the compressed air was 10 lb./inch.² in the majority of experiments. The results, shown in graphs, determined the dimensions of the final design built of glass and giving a 3 cm. pressure drop with a delivery pressure of 5-6 lb. In an appendix the manufacture of an air ejector from standard copper alloy fittings is described which may be preferred to glass for safety reasons.

Sommaire : Détails sur la construction d'un éjecteur à air pour pomper des coffrets secs.

Report by J. R. Catlin L. Coward D. Darling W. T. Spragg & C. G. Webb *A.E.R.E. Report* No. C/M 152 1952

A New Design of Filter for Dry-Boxes, Type D/47

46/II

United Kingdom. A filter is described which is employed in connection with dry-boxes used for chemical and physical operations carried out at sub-atmospheric pressures. The filter is to intercept any radio active or toxic material which otherwise might reach the pump and contaminate the oil. It consists of a circular metal cannister, 6½ inch. diameter and 3½ inch. long, supported by a special fitting attached to the dry-box and has been fabricated from components used in respirators of certain types. The filter is made up of three parts, a layer of activated charcoal 1 inch. deep for the removal of acid vapours, a mechanical filter of wool and asbestos and a second, high efficiency mechanical filter of resin-impregnated wool. Tests with methylene blue using particles of mass median diameter of less than 0.5 micron showed an overall penetration of 0.0001%. Normally the filter should be effective for about six months. Its performance can be currently checked by measuring the pressure drop across the filter with a simple manometer. In the case of a new filter the pressure drop should be approximately 1.5 inch. water gauge at a flow of 3 ft. ³/min.

Sommaire : La construction d'un filtre est décrite, lequel serait utilisé en connection avec des coffrets secs, lequel doit empêcher le passage de matière radio-active dans le pompe à vide.

Report by W. G. Busbridge *A.E.R.E. Report* No. HP/M.34 1952

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21 — GAUGES — 21

A Recording Mercurial Manometer for the Pressure Range 0–760 mm. of Mercury 47/II

United States. A simple and reliable recording manometer was required in a series of experiments concerned with reaction phenomena between moderately active metals and water vapour which were to be followed by observing the increase of pressure caused by the hydrogen evolved. The pressure range to be covered was 0–600mm. Hg. As no suitable instrument was available commercially a new design was developed specially for the purpose. It consisted of a large bulb mercury manometer with a 6mm. diameter Pyrex tube 85 cm. long carrying in the centre a resistance wire (Nichrome V No. 24) which was part of a d.c. bridge circuit. At zero pressure the full length of the wire was included in the bridge circuit but as the pressure rose the effective length of the wire was gradually reduced. The change in resistance over the full pressure range was 4 ohm. The bridge was supplied by a stabilised power unit of 300 V. One leg consisted of a current limiting resistor and the manometer wire and the other leg of another current limiting resistor in series with a fixed resistor. The latter two determined the maximum zeroing sensitivity for the recording potentiometer incorporated in the circuit as the balance indicating device. In the particular case the following values were chosen : The potentiometer had a range of 0–50 mV, the total current drawn was 15 mA, the manometer wire (full length) equalled 4 ohm, the current limiting resistor in the manometer leg = 24,000 ohm, the other current limiting resistor = 150,000 ohm and the fixed resistor = 25 ohm. A current of 12.5 mA passed through the manometer leg and when the effective length of the manometer wire was shortened the current changed in the proportion of 1 part in 6,000 over the entire pressure range. The current in the other leg was 2 mA which facilitated zeroing the pen of the recording potentiometer when the pressure was zero. The upper limit, 760 mm. pressure, was recorded at 50 mV. With slight modifications a manometer of this design can be used as a pressure control device.

Article by H. J Svec and D. S. Gibbs *Rev. Sci. Instrum.* **24**, March 1953 202-204

Sommaire : Un manomètre à colonne de mercure a été construit, pour l'enrégistrement continu des pressions entre 0–760mm. Hg pour étudier la réaction entre les métaux et la vapeur d'eau.

Temperature-Stable Capacitance Pressure Gauges 48/II

United Kingdom. The author, engaged in the construction of capacitance pressure gauges, found that instabilities observed were predominantly due to temperature changes. Generally, the characteristics of the gauge such as sensitivity, linearity and frequency response have been explored very fully but little has been published on temperature effects. In the present paper equations developed by other workers have been extended to account for the above-mentioned observations. The total capacity of a pressure gauge can be considered to be made up of three components : (*a*) The capacitance of the electrode to the diaphragm at zero applied pressure, (*b*) the ' stray ' capacitances and (*c*) the change in capacitance due to a given applied pressure. (*c*) is determined by the nature of the diaphragm. A formula is given for the overall temperature coefficient of the total capacitance which serves as an indication of stability. The temperature coefficient of (*a*), the diaphragm capacitance, is the most important. Its value, in turn, is governed by three sets of conditions : Changes in area of the electrode and changes of gap size resulting from differential expansion, constraints acting in the diaphragm causing distortion and non-uniformity of the temperature in the gauge due, for example, to hot fluid impinging on one side. A theoretical treatment of these conditions, given in appendices to the paper, yields formulae useful for the assessment of major design features. In the second part of the paper the practical design of a capacitance gauge developed on the basis of the results of the theoretical considerations is described. The gauge was built for the pressure range 0–10mm. of water. Its capacitance was fixed by design considerations at 100 pF. A stretched membrane made from 0.002 inch. steel shim was used as the diaphragm which had a sensitivity of 0.23 pF. per mm. of water and a total capacitance of 80 pF. Its overall temperature coefficient in the range of 10°C. around room temperature was only –100 p.p.m/°C.

Article by D. C. Pressey *J. Sci. Instrum.* **30**, Jan. 1953 20-24

Sommaire : L'effet des changements de température sur les jauges de pression à capacitance est discuté théoriquement et la construction d'une jauge à coefficient de température sur la capacitance, faible, est décrite.

A Diaphragm-Type Capacitance-Type Micromanometer for Very Low Differential Pressures 49/II

United States. Until recently the limit of resolution for diaphragm micromanometers was 1 mm. Hg but Sibeler and Cordero have lately designed an instrument lowering the limit of resolution to 0.1 micron Hg. A similar instrument is described and illustrated in this report. It contains a corrugated diaphragm 0.001 inch. thick and 3 inch. diameter wide and is combined with a capacitance-type pick up and resonant-bridge carrier system of 0.005 micro-microfarad sensitivity. Details of the calibration method employed are given. An Ascot Casella micromanometer was used for the purpose and the required pressure changes were effected with the help of a hypodermic syringe. The results of experimental calibrations are shown in 2 diagrams; one applying to an instrument with an electrode spacing of 0.017 inch, and the other to an electrode spacing of 0.0035 inch. If one side of the diaphragm is joined to a vacuum system the instrument can be used as an absolute pressure gauge and might provide a means of calibrating ionisation gauges.

Report by T. A. Perls, W. H. Kaechele & D. S. Goalwin *N.B.S. Report* No. 2165 January 1953 7 pp.

Sommaire : Description d'un micromanomètre à diaphragme du type à capacitance pour des basses pressions differentielles et convenable pour la mesure absolue de pressions de l'ordre de 0.001mm. Hg.

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Cold Cathode Ionisation Gauges for the Measurement of Low Pressures

50/II

United Kingdom. Cold cathode ionisation gauges in which the discharge is maintained in an axial magnetic field provide a convenient and rapid means of measuring pressure in the range 10^{-2} to 10^{-7}mm. Hg. These instruments are simple to make and operate and it is virtually impossible to damage them. With such a gauge, measurement of pressure is effected by a robust pivot-meter which measures a current of ions formed by electron bombardment. Primary electrons are released by cold emission from a pair of cathodes and many secondary electrons are generated by the ions themselves. The magnetic field causes the electrons to move in tight spirals to and from between the two cathode plates. In this way the number of ions per electron, between initial emission and eventual escape from the discharge to the anode, is much increased. A review is given of the construction characteristics and performance of various types of cold cathode gauge. The influence is reported of size and shape of the electrodes of applied voltage, of magnetic field and of the nature of the gas. Reference is made to the performance at relatively high pressures (10^{-2} to 10^{-3}mm. Hg). Attention is drawn to the generation of radio frequencies by cold cathode gauges.

(*Authors*)

Article by G. K. T. Conn & H. N. Daglish *Vacuum* 3, Jan. 1953 24-34

Sommaire : Revue de la construction, des caractéristiques et performances des differents types de jauges à cathode froide. Il est rendu compte d'un nombre de facteurs tels que les dimensions et la forme des électrodes, le voltage appliqué etc.

A Degassable Penning Gauge

51/II

France. Generally the operation of the Penning gauge is satisfactory, but degassing is still difficult. Normally high frequency heating is employed in this process. The equipment for this is expensive and requires that the gauge is in a readily accessible position and that the magnet has to be removed. Also the temperatures obtained are not necessarily uniform over the whole area of the electrodes. Of the two other heating methods which might be used for degassing, namely resistance heating and heating by electron bombardment, the latter proved impracticable in experiments carried out by the author. But a modification of the design of the electrodes is presented which facilitates resistance-heating of the electrodes. Both electrodes are fabricated from molybdenum sheet 1/10th mm. thick with slots milled into it in order to increase the resistance of the material to the passage of the current. The anode is cylindrical and the cathodes are made from flats. A diagram is shown of the circuit. The heating current is taken from a transformer with 2 low-tension windings of 3 V 15 A each, one circuit heating the anode and the other heating the two cathodes. A three-way switch is provided with a special degassing position.

Article by A. Bobenrieth *Le Vide* 8, March 1953 1302-1304

Sommaire : Description d'une jauge de Penning dont les éléctrodes sont construites pour faciliter le dégasage par résistance chauffante.

A Simplified Ionisation Gauge Circuit

52/II

Canada. The stabilising circuits of the conventional ionisation gauge are normally based on grid current stabilisation. This involves the provision of extra power supplies which is costly. Sibati, Tuzi and Kumagai recently described a gauge (see Vol. II Abstract No. 57/II) fitted with a circuit which provided for the measurement of the ratio of electron to ion current. This eliminated all variations and instabilities caused by effects acting on both currents simultaneously. The author describes a further development of that circuit. A 2-ohm rheostat is used to adjust the filament current until the normal grid current is flowing. The latter passes through the meter shunt and then through a resistor producing a grid voltage in one of two pentodes provided in place of the batteries employed by Sibati *et al.* for the plate voltage. Subsequently the meter switch is placed in the measuring position, the range switch and the resistor are placed on zero and the circuit balanced with the help of a 5 K potentiometer as indicated by a zero reading. From the values of the resistor settings required the ratio of plate to grid current can be determined and from the manufacturer's data the controls can be calibrated directly in pressure units. Details of the degassing procedure are given. A detailed diagram of the circuit is shown. The use of the gauge for leak detection purposes is described. The entire instrument can be built for about $50 as against $200 required for the instrument with the conventional circuit.

Article by F. J. Fitz Osborne *Canad. J. Phys.* 31, Jan. 1953 11-14

Sommaire : Déscription du circuit d'une jauge à ionisation qui représente un raffinement de celle décrite dans Vol. II resumé No. 57/II.

22 — MEASURING PLANT (VACUUM) — 22

Field Emission Microscopy

53/II

United States. Research of metal surfaces aims at an analysis of surface structure down to atomic dimensions. The grain structure and shape of powder particles can be investigated in the light and electron microscope, the latter shows films of a thickness down to 20 Å and has a resolving power about the same in all three dimensions. The electron diffraction method gives information on the average surface structure some 20–50 Å above the surface. Only the field emission microscope with an efficiency 10 times that of the electron microscope in depth shows the presence of monoatomic layers and thus facilitates the study of surface migration, wetting,

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oxidation and the like phenomena in the layer, where they occur. Basically the instrument consists of an evacuated envelope fitted with a needle-shaped cathode, in front of a ring-shaped anode, pointing to a fluorescent screen. Applying up to 15,000 V produces a field of up to 50,000,000 V/cm. causing electrons to be emitted from the cathode tip by the tunnel effect which hit the screen and yield an enlarged projection picture of the tip surface. With a tip radius of 10^{-5}cm. magnifications up to 500,000 can be obtained. The high current density of the emission up to 10^6 A/cm.^{2} ensures a bright picture which can be photographed with a motion picture camera. The resolving power of the instrument is 10–20 Å. So far tungsten, molybdenum, tantalum, nickel, platinum, iridium, zirconium, iron, copper and silver have been used for the tips, normally consisting of a single crystal which, as long as it is bare, shows a symmetric pattern. The radius of the tip increases continuously during operation and can be determined by calculations if the anode voltage required to produce a certain emission current is known. As the work function of the clean tip is greatly influenced by the presence of even a small fraction of a monoatomic layer of foreign matter the instrument is particularly suitable for the study of adsorption phenomena, chemical compounding and evaporation mechanisms. The emission is independent of temperature. Thus by cooling the tube reactions such as the formation of multi-layers of permanent gases occurring at 80° K can be observed. A very high vacuum is required for these experiments. Gases, like oxygen tend to stick to a clean surface on contact and even at a pressure of 10^{-6} mm. Hg some 10^{14} molecules are still hitting 1 cm.^{2} of a surface per second. This facilitates the formation of a monolayer in a few seconds. For this reason baking of the tube is indispensable. The adsorption of oxygen by tungsten has been investigated. Oxygen layers decrease the emission appreciably. At room temperature two layers were adsorbed on top of each other. The second could be removed by heat treatment at 1,100° K in high vacuum. The first appeared to form a surface compound but was successfully reduced by hydrogen at 10^{-3} mm. Hg, the tube being immersed in liquid air, in the temperature range from 1,200–1,500° K with the result that a perfectly clean tungsten surface was obtained in a few seconds. When a very thin layer (less than one monolayer) is adsorbed from material consisting of large atoms or molecules blurred dots appear in the picture which in the case of barium could be identified as individual atoms of approx. 20 Å diameter. If deposited in a protruding position, atoms of 3–5 Å can be made visible. Adsorbed atoms can be caused to leave the surface in the form of ions by applying a very high positive field strength to the tip. The ions travel on the same path as the electrons but their tangential velocity is very much smaller, reducing the scattering effect observed with electrons and resulting in an improvement of the resolving power to approximately 3 Å. This observation has led to the development of the field ion microscope. The application of the latter is still limited because field strengths up to 300,000,000 V/cm. are required exerting a mechanical force of approximately 600,000 lb./inch.^{2} very nearly the limit of the tensile strength of tungsten, let alone that of any other metal.

Sommaire : Compte rendu détaillé sur la construction, performance et les avantages offerts par la microscopie à champ d'émission.

Paper by E. W. Mueller *Mod. Res. Techniques Phys. Metallurgy* Conference Oct. 1952 33-50

Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy

54/II

United States. It is known that a soluble protein if applied to a point on an aqueous surface will spread across the surface in the form of an insoluble monolayer effecting at the same time a uniform distribution of any particulate matter that may be embedded in the monolayer. This effect can be used for the preparation of specimens such as bacteria and viruses for examination in the light and electron microscope. In the method described an 0.05% solution of bovine serum albumin is used as the suspending medium. The solution containing the particles is spread over the surface of distilled water held in stainless steel trays, 0.7 cm. deep and of an area of 21.5 × 6.5 cm.^{2}; 10 mg./1. zinc chloride is added to the water in order to speed up the spreading of the protein solution across the surface. When transferring the monolayers to a solid surface it is necessary that the film pressure is kept constant. This can be achieved with the help of material such as piston oil tri-m-cresyl phosphate with a constant spreading pressure of about 9 dynes per cm. The piston oil is applied to the clean liquid surface inside a loop formed by a single strand nylon thread and transmits its pressure across the thread. If the counter pressure due to the pressure of the monolayer is equal to the pressure of the oil the loop will assume an irregular shape, should the monolayer encroach on the area covered by the oil. This can be rectified by adjusting the position of a moveable barrier across the trays. This method has the additional advantage that the total area covered by the monolayer at constant pressure can be determined. Loss of particles below the surface can be prevented by placing a glass slide across the tray immersed in the liquid. The slide carries three or four wire mesh screens, covered in the usual way with a collodion film. Details of the preparation of specimens of *E.coli*, *S.marcescens* and *type III pneumococci* are given and illustrations are shown demonstrating the quality of micrographs obtained from specimens prepared by the method described.

Sommaire : Rapport sur les méthodes améliorées de préparation de specimen biologiques pour l'examen au microscope électronique.

Article by R. E. Hartman, T. D. Green, J. B Bateman, C. A. Senseney & G. E. Hess *J. Appl. Phys.* 24, Jan. 1953 90-92

A Method of Freeze Drying for Electron Microscopy

55/II

United States. The ordinary methods employed in the preparation of biological specimens for examination in the electron microscope are known to result in deformation of the material caused by surface tension, in particular, during the final stages of drying. To overcome this, a freeze drying method has been applied as described by Wyckoff, but the results were not satisfactory. Anderson developed the critical point method where the specimen material is caused to ' dry ' out of CO_2 at a temperature above its critical point. Good results were obtained but the method has several drawbacks, for instance the specimen can only be observed

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in a chemically fixed state and cannot be shadowed as it is permanently trapped between two films of collodion. In order to achieve success with the freeze drying technique in this particular application freezing of the aqueous suspension must be effected rapidly, the ice must sublime at a temperature well below the freezing point of any eutetic concentration which may develop at this stage, the temperature of the specimen material must not rise before the ice has sublimed and should be higher than room temperature when it is exposed to air. Finally, after drying the material should be well spread out. Examining the freeze drying method currently practised the author came to the conclusion that its major defect is the comparatively high temperature of the specimen preventing proper sublimation. An improved method is described which provides for droplets of the suspension to be sprayed onto a collodion-coated copper support, the temperature of which can be controlled. and subliming the ice at a well defined low temperature. A diagram is shown of the arrangement. The copper block covered with a collodion film is inserted into a glass tube and fixed to the bottom with vacuum grease. The tube is immersed in liquid nitrogen for about 5 minutes until the copper support has reached equilibrium temperature. Then 0.01 ml. of the suspension is sprayed with a nebuliser of normal commercial type onto the collodion film, the tube, kept at –50°C. is closed and evacuated to less than 10^{-4}mm. Hg *via* a liquid nitrogen trap. Sublimation is completed after 15 minutes. The tube is then transferred for 10 minutes to a bath at 70°C. raising the temperature of the copper surface to 50°C. The vacuum is broken and the copper support removed to a shadowing unit. After shadowing the collodion film is detached from the copper support and the specimen introduced into the electron microscope. An alternative to this method provides for the droplets to be frozen in flight. This will prevent contact of the suspended biological material with the wet collodion substrate but the rate of freezing is not necessarily as rapid as in the former method. The results of the new methods are compared with the critical point method. Hemolysed human erythrocyte, known to react to the slightest force of surface tension, was used for the tests, *i.e.* the same material as that used by Anderson. Fixed and unfixed cells were prepared. The fixed cells gave better results. Their three-dimensional structure compared favourably with that produced by the critical point technique. Measurements of the frozen dried droplets produced by this method indicated a droplet volume of about 10^{-9} ml., an area of 4×10^{-6}cm.^{2} and a resultant thickness of 2.5×10^{-4}cm. The theoretical rate of change of temperature in the experimental conditions was calculated to be 4×10^6 degrees/sec. Its practical value can be assumed to be 2×10^5 degrees/sec., *i.e.* 10^4 times greater than that obtained in the old method. Suitable suspension media (of satisfactory volatility under the conditions of the process) are 2% solutions of ammonium succinate or ammonium benzoate, at pH 7.0.

Article by R. C. Williams *Exp. Cell Res.* 4, Feb. 1953 188-201

Sommaire : Description des raffinements de la technique de lyophilisation employée pour la préparation de spécimen biologiques pour l'étude au microscope électronique, qui élimine les effets de la tension superficielle du spécimen.

The Formation of Adsorbed Layers in Vacuum Chambers and Their Detection by Electrons 56/II

See Abstract No. : 77/I

Specimen Changes and Electron Optical System in Electron Microscopes 57/II

Switzerland. The number of cases where the results of examinations in the electron microscope are criticised on the grounds that the specimen may have shrunk or suffered deformation and structural changes during exposure to the beam is on the increase. The author carried out methodic experiments in order to explore these conditions. A Trüb-Täuber Type KM electron microscope was used fitted with a cold-cathode Induni-type source. The source voltage was kept constant at 45 kV throughout the experiments. The magnetic condenser lens was 21 cm. away from the cathode and 9 cm. away from the specimen. It had an aperture of 0.3 mm. diameter. The sectional area of the beam hitting the specimen could be limited to the size of the hot spot of the source, which was originally 0.01 mm. in diameter growing to 0.03 mm. diameter in about 1,000 hours. Magnification was 10,000×. A quick acting focussing device was used. It works on the principle that 3 pictures of the specimen area to be examined are produced initially and have to be merged into one. In preliminary experiments the two main factors causing changes in a specimen were established to be :—The intensity of the radiation and the exposure time, and to a lesser degree the effects of the contrast diaphragm. In order to determine permissible values for the former, cellulose specimens on collodion films shadowed with gold, palladium and platinum were exposed to the beam in the instrument for various periods. The energy

Table I

Manipulation	*Density Charge W/cm.²*	*Time Sec.*	*Energy Density of Irradiation W sec./cm.²*
Observation 200x	1	15	15
Observation 10,000x	200	15	1,000
Adjustment	15	10	150
Rough Focussing ...	500	5	2,500
Fine Focussing ...	2,000	3	6,000
Taking of the Picture	15	15	225
Total Energy Density		~ 60	~10,000

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density of irradiation, defined as the product of the energy density of the beam and the exposure time, served as a yardstick of comparison. The minimum energy density of irradiation is determined by the time required for the various manipulations as per timetable shown above (emission current: 0.5 m.A). After 40–60 seconds a certain contrast reduction was noticeable and this became gradually more pronounced as irradiation continued. At the same time the specimen appeared to become flatter. The use of a contrast diaphragm made no difference to this change in appearance, a sure indication that a structural change had taken place. The use of the contrast diaphragm is discouraged as it will increase contrast where some deposit on the specimen causes contrast reduction and thus it might produce misleading results. Further, the experiments proved that the permissible energy density of irradiation steadily decreases as the nature of the specimen material becomes more delicate. The 10,000 Watt sec./cm.^{2} shown in Table I can already be considered excessive. As one means of cutting down exposure to full irradiation energy it is suggested to adjust the focus on a spot immediately adjacent to the area of the specimen to be photographed and to switch the beam back to that area after focussing manipulations are completed. This results in a considerable gain as shown below (emission current 0.2 mA). Illustrations show the improved quality of the micrographs obtained by the

Table II

Manipulation	*Density Charge W/cm²*	*Time*	*Energy Density of Irradiation W sec./cm²*
Observation 200x	0.5	20	10
Surveying 10,000x	250	—	—
Observation 10,000x	250	5	1,250
Adjustment	15	10	150
Rough Focussing ...	250	—	—
Fine Focussing ...	1,000	—	—
Change over to Specimen Proper ...	15	10	150
Focussing Control ...	1,000	0.5	500
Taking of the Picture	15	4	60
Total Energy Density			~ 2,000 W sec./cm²

suggested method. It is stressed that the experimental results are based on the provision of a ' clean ' vacuum. Any contamination of the specimen will substantially reduce the permissible limit of energy density applicable, an effect which is quite independent of the type of pump used.

Sommaire : Recherches sur les causes des changements se produisant dans des specimens examinés au microscope électronique et conseils pour éviter de tels changements.

Article by L. Wegmann *Optik* 10, Issue 1/3, 1953 44-59

Improvements in Apparatus for Introducing Specimens into an Evacuated Chamber

58/II

United Kingdom. Normally, when changing a specimen the vacuum in the electron microscope or diffraction camera is broken resulting in the need for repumping the chamber. This takes time. It is proposed therefore to introduce the specimen through an airlock which prevents large volumes of air from entering the chamber. The device consists of a rod sliding with an airtight fit in a tube, the tube passing through the wall of the chamber to atmosphere. The rod has a series of holes near the inner end to accommodate the specimen. In order to load or unload the specimen the rod is pulled outwards. At first, it slides within the tube until the holes are covered, and an extension at the inner end of the rod abuts against the tube. Then, both rod and tube slide as a unit through the opening in the chamber wall until a snap-ring fitted to the extreme end of the rod hits the chamber wall. The tube is now moved forward to expose the holes in the rod. The only air introduced during the manipulations is that caught in the specimen holes—a quantity so small that it can be pumped away in a few seconds. By the provision of metal bellows in a suitable position proper scanning movement of the specimen in the chamber is facilitated.

Sommaire : Description de la construction d'un appareil facilitant l'introduction de specimen dans le chambre évacuée d'un dispositif de mesure du vide.

R. S. Page & Metropolitan-Vickers Electrical Co. Ltd. *Brit. Pat.* 682,530

A Vacuum Lock for Routine Solid Analyses with a Mass Spectrometer

59/II

United States. The use of the mass spectrometer for the analysis of solids is still restricted, largely for the reason that the process is slow. The introduction or changing of samples takes no longer than five minutes, but the vacuum has to be broken and re-pumping takes up to two hours. Assuming the analysis itself takes one hour, no more than three samples can be dealt with in one day. The article describes an airlock designed for incorporation in a mass spectrometer. It consists of a long shaft in three sections. Two carry a sample each and are called ' carriages '. The third, placed between the carriages, is a piece of blank rod. The rod sections are moved through a channel extending through the source region of the instrument like slides in a slide projector. The rods consist of 7/16th inch tubing and the clearance between the rods and the channel is approximately 0.003 inch. The tubes are cut open in the place where the sample is situated carrying with it about 100 cc. of air. As the sample moves along the channel it passes three outlets in the channel, the first two of which are connected to rotary pumps reducing the pressure to 1 mm. and 0.01 mm. Hg respectively, and

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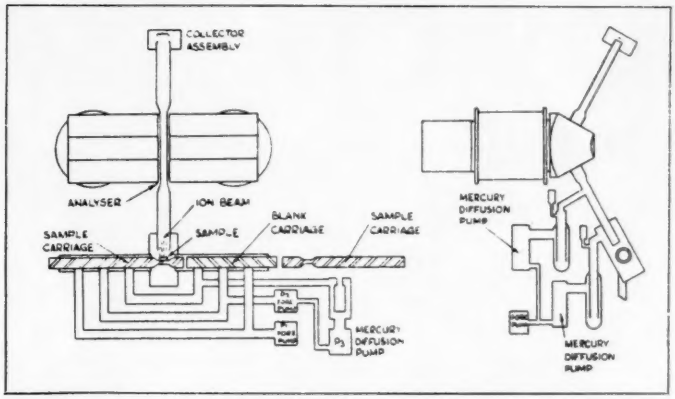

the third, the nearest to the source chamber, to a diffusion pump with an ultimate pressure of approximately 2×10^{-6}mm. Hg. At each stage the required low pressure is reached in about 10–20 seconds. A double 'O'-ring seal at the end of the channel provides a seal against the in-leakage of atmospheric air. The ion source is designed on the lines of the surface-ionisation type proposed by Inghram and Hess. The anode plate with the filament holder and filament (supporting the sample) as well as a drawing-out electrode are incorporated in the carriage. The ions emitted from the filament are accelerated to two defining slits at ground potential fitted to the end of the (fixed) spectrometer tube. Provisions are made to shield the region between the drawing-out plate and the first ground slit from the grounded side sections of the carriage. The lock described fits an Inghram and Hess mass spectrometer of 12 inch. radius of curvature, 60° sector type. Differential pumping of the source region is used. The use of the lock practically eliminates all pumping down times. The pressure in the analyser is only slightly affected by the sample change as the defining slit is the only communication with the source region and the slit has a dimension of 0.008 inch. × 9/16th inch. After ten minutes the required operational pressure is re-established. The use of the device facilitated six runs per eight-hour day.

Sommaire : Il a été établi un système d'écluse pour l'introduction de matières solides dans l'analyseur d'un spectromètre de masse, sans deteriorer le vide.

Article by C. M. Stevens *Rev. Sci. Instrum.* **24**, Feb. 1953 148-151

Apparatus for Supplying Charge Material to Mass Spectrometers

60/II

United States. A Vacuum lock system is described for the continuous or intermittent introduction of solid or liquid materials into the ionising region of a mass spectrometer without breaking the vacuum. The housing of the lock system is attached to the instrument either along with, or perpendicular to, the axis of the analyser tube and contains three comparatively large cavities connected with each other by a channel 0.1 inch wide and 0.015 inch high. Each cavity is connected to a vacuum pump, the first to a mechanical pump reducing the pressure in the cavity to about 1mm. Hg, the second to a diffusion pump reducing the pressure in the second cavity to about 1 micron Hg and the third to a second diffusion pump with the first diffusion pump as a backing pump. Here the pressure is reduced to 10^{-6}mm. Hg. This pump may serve for the exhaustion of the analyser chamber as a whole. The means of introducing the samples is a metal tape, preferably of tungsten, tantalum or Nichrome, 0.0063 inch thick and 0.097 inch wide run off and returned to drums positioned outside the housing which travels at a speed of 1 inch per second. A second tape travelling at the same speed is brought into contact with the first during its passage through the above-mentioned channel in order to protect the samples but is diverted on entry into the instrument chamber. The device can be operated in conjunction with any conventional ionising means but a design specially adapted to this arrangement is described. The version for the intermittent introduction of samples consists of a similar housing provided with three major cavities as before and with a similar pumping arrangement. The sample is introduced at the top of a rod which is pushed through the channel and in the operational position is exposed to the ionising source permanently fixed in the spectrometer head. When the rod is withdrawn each cavity is sealed automatically by a valve to avoid in-leakage of atmospheric air. The valves are re-opened by a control rod moving at the same speed as the probe, causing the sealed bellows-type valve housings to move outwards by a camming action.

Sommaire : Système d'écluse convenant à l'introduction d'échantillons solides ou liquides dans la tête d'un spectromètre de masse sans interrompre le vide. La partie essentielle étant un tapis roulant.

V. L. Parsegian, C. M. Stevens & Atomic Energy Commission *U.S. Pat.* 2,618,750

Vacuum Bakeout Ovens

61/II

United States. A production arrangement for the cleaning and degassing of mass spectrometer components is described briefly. The essential piece of equipment is a 2.5 kW. vacuum oven which is mounted directly on the mouth of a diffusion pump with its controls on an adjacent table. A Pyrex bell jar constituting the oven

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chamber carries Nichrome ribbon serving as the heating element on the outside and is protected by a metal hood. Both bell jar and hood are counterbalanced for ease of handling. The parts to be treated are first washed in a warm detergent solution then rinsed in water, alcohol, benzene and ether and finally dried by radiant heat. They are then transferred by tweezers to the oven and baked for approximately three hours at 400°C. at a pressure of 2×10^{-6}mm. Hg. After baking the parts are placed in helium-filled containers for storage or transport.

Sommaire : L'equipment et le dispositif de production pour le degazage et le nettoyage des éléments d'un spectromètre de masse sont brèvement dècrits.

Note by Anon. *Electronics* **26**, Feb. 1953 276-278

Electron Diffraction Investigation of Polished Surfaces — **62/II**

See Abstract No. : 121/I

26 — MATERIALS — 26

Improvements i.o.r.t. a Process and Apparatus for Evaporating Liquids — **63/II**

See Abstract No. : 77/III

Industrial Applications of Activated Alumina — **64/II**

United Kingdom. Activated alumina collects the moisture present in the surrounding atmosphere by physical adsorption as distinct from desiccants which absorb the moisture and combine chemically with it. After use the moisture held in the pores can be removed from the adsorbent by heating. In this manner the desiccant can be regenerated and used indefinitely, whereas the chemical desiccant can only be used once. Activated alumina is a hard micro-crystalline material which absorbs up to 35% of its own weight of water in equilibrium at 100% relative humidity and 15°C. For regeneration it has to be heated to a temperature between 150°–250°C. Performance is best at low temperatures. As the latent heat of condensation of the water vapour raises the temperature of the alumina bed the absorbent should be kept cool. Contamination by oil vapours should be avoided. Oil absorbed by the alumina carbonises during regeneration, and clogs the pores. In practice, the desiccant is placed in towers of a height three to four times their diameter and the gas to be dried is passed in a downward direction. For regeneration the flow is reversed. Various industrial applications of activated alumina are mentioned, for example, its uses for the protection of the machinery of laid-up ships by a special method employed in the U.S.A. In pellet form it can be added to solids for protection during transit to be separated out again before the material in question is used. Activated alumina is an effective catalyst and is frequently employed in this capacity. By adding special catalytic agents to the alumina during manufacture its catalytic performance can be enhanced and made to last longer.

Sommaire : Discussion de l'emploi d'alumine activée comme dessiccant et catalyseur.

Article by J. H. Harwood *Times Rev. Industr.* 7, 72 (New Series), Jan. 1953 p. 29

Evaluation of Vacuum Gasket Materials — **65/II**

See Abstract No. : 71/II

Thermal Degradation of Polystyrene — **66/II**

See Abstract No. : 115/I

Behaviour of Refractory Oxides in Contact with Metals at High Temperatures — **67/II**

United States. Techniques employed in the field of nuclear energy and supersonic flight often provide for the use of metals in contact with refractories at elevated temperatures. The behaviour of a number of metal-refractory combinations have been studied in preliminary experiments, details of which are given in this paper. A diagrammatic sketch of the furnace unit used in the experiments is shown. The vacuum was produced by an oil diffusion pump backed by a mechanical fore pump. The induction heating system was supplied by a 100 h.p. generator of 50 kW, 9,600 cycle (Tocco unit). The general arrangement of the heating system was the same as that described by Johnson (*J. Amer. Ceramic Soc.*, **32**, 1949, 316 and **33**, 1950, 168). The gas purifier train contained a cold trap to remove the water vapour, a chamber with magnesium chips heated at 700°C. to remove the oxygen and various calcium sulphate and phosphorous pentoxide driers in series to remove the last traces of moisture from the gases introduced into the furnace. The refractory oxides examined were Al_2O_3, MgO, BeO, ZrO_2, TiO_2, ThO_2, all of the highest purity commercially available. The oxides were used in the form of sintered and polished discs, a reaction and a cover disc of each oxide, 0.75 inch in diameter and 0.25 inch thick. The following metals were investigated : Titanium, beryllium, silicon, niobium, nickel, molybdenum and zirconium. Samples of the metals in powder or pellet form were placed between a pair of discs and fired in the furnace at 1,400, 1,600 and 1,800°C. in a helium atmosphere of 30 cm. Hg. Before firing the furnace was

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Reaction Summary Chart[a]

	Mo				*Ti*			
	Temp. °C. 1400	1600	1800	*New Products*	*Temp. °C.* 1400	1600	1800	*New Products*
Al_2O_3	NR	NR	NR	—	NR	SR	*VR*	TiO_2
BeO	•	NR	NR	—	•	SR	*VR*	TiO_2
MgO	NR	NR	NR	—	SR	SR	*VR*	$MgO.TiO_2$
ThO_2	•	NR	NR	—	•	NR	*NR*	TiO_2
TiO_2	NR	NR	••	—	NR	SR	••	?
ZrO_2	NR	NR	NR	—	NR	SR	*VR*	TiO; s.s. of Ti
	Be				*Si*			
	Temp. °C. 1400	1600	1800	*New Products*	*Temp. °C* 1400	1600	1800	*New Products*
Al_2O_3	*NR*	*NR*	*SR*	$BeO.Al_2O_3$	SR	*VR*	O	SiO_2; $3Al_2O_3.2SiO_2$
BeO	•	*SR*	•	—	•	*VR*	•	—
MgO	*SR*	*VR*	O	BeO	SR	*VR*	O	$2MgO.SiO_2$
ThO_2	•	*SR*	•	—	•	*VR*	•	—
TiO_2	*SR*	*VR*	••	?	SR	*VR*	••	SiO_2
ZrO_2	*NR*	*SR*	O	BeO	NR	*VR*	O	$ZrO_2.SiO_2$

[a] Code : NR = no perceptible reaction; SR = slight reaction as shown by surface discolouration and penetration; VR = violent reaction as shown by presence of a new phase or surface corrosion; O = omitted because of excessive volatilisation of the metal at 1800°C.; •• = omitted because TiO_2 fused below 1800°C.; • = omitted because only few specimens were available; ? = X-ray lines show unidentified reaction product; s.s. = solid solution shown by *d*-line shift in x-ray study.
[b] Italics indicates molten metal in contact with the oxide.

evacuated, hydrogen admitted and the furnace heated to 600°C. in order to remove all residual oxygen. The operational temperature, attained gradually at a rate of 20°C. per minute, was held for 15 minutes before the power was shut off. The treated specimens were examined visually and metallographically. The results of the experiments are shown in a table, partly reproduced above. At temperatures below 1,400°C. the oxides were little affected but at higher temperatures reaction became more pronounced. Nickel and molybdenum were stable in all tests but the other oxides showed decreased stability in the following order :—ThO_2, ZrO_2, Al_2O_3, BeO, MgO, TiO_2. Beryllium and silicon reacted with all oxides at high temperatures. Zirconium and titanium reacted with the oxides probably by some form of oxygen exchange mechanism due to their high oxygen solubility.

Sommaire : Recherches prélimaires sur le comportement d'oxydes réfractaires, tels que MgO, TiO_2, en présence de métaux tels que Ti, Si, Zr aux températures élevées.

Article by G. Economos *Ind Engng. Chem.* 45, Feb. 1953 458-459

Plating as an Aid in the Brazing of Stainless Steel 68/II

See Abstract No. : 95/I

28 — APPARATUS COMPONENTS — 28

A Controlled Gas Leak 69/II

See Abstract No. : 82/I

A New Design of a Filter for Dry-Boxes. Type D/47 70/II

See Abstract No. : 46/II

Evaluation of Vacuum Gasket Materials 71/II

United States. The evaluation of the vacuum properties of materials attempted in the investigation reported here is restricted to the determination of the relative order of values for the outgassing rates and vapour pressures of the materials. In the experiments, the pump-down characteristics of a given vacuum system were established by plotting a time-pressure curve and comparing these values with a second pump-down curve obtained after inserting a sample of the material to be tested into the system. A small glass chamber fitted with an ionisation gauge and served by a 3-stage fractionating glass diffusion pump backed by a rotary pump was employed. All measurements were taken at room temperature. No vapour traps were included. The samples were cut to give comparable exposed surfaces of approximately 2 sq. inch. The time-table of the procedure is given as follows :—Insert sample and start the rotary pump; 20 minutes later, start the diffusion pump; 10 minutes later, switch on the gauge; take readings every 5 minutes for a half-hour;

II — Vacuum Apparatus and Auxiliaries — II
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Abstract No. and References

take ½-hourly readings for the next 2–3 hours; take a final reading after 24 hours. The materials tested were:— Teflon, Silicone SR–1060, Myvaseal, Silicone SR–1080, Koroseal, Hycar OR–15, Neoprene, Tygon and Garlock 7986. The results are shown in graphs. A table reproduced below gives the relative vapour pressures in an

1. Teflon	6. Hycar OR–15
2. Silicone SR–1060	7. Neoprene
3. Myvaseal	8. Tygon
4. Silicone SR–1080	9. Garlock 7986
5. Koroseal	

increasing order based on the pressure after 6½ hours exposure but these values may not be quite accurate, as the response of the Ionisation gauge to the various vapours was not known.

Sommaire : Une méthode déstinée à déterminer les performances de matériaux normalement utilisées comme joints dans systèmes à vide, est discutées.

Report by L. E. Normand & W. R Rathkamp Oak Ridge National Laboratory 7-12 Area, Oak Ridge, Tenn. U.S.A.

Vacuum ' O '-Ring Seals — 72/II

United Kingdom. Normally one of the two mating faces of an ' O '-ring seal carries a machined groove to hold the ' O '-ring. With grooves of conventional cross section it was found difficult to obtain adequate compression and retention of the ' O '-ring in operation. A new design of an ' O '-ring seal has been developed therefore which provides for the groove to be of a trapezium cross section. Design details of the new seal are given.

Sommaire : Description d'un nouveau joint torique ayant un logement à section trapézoidale.

Letter by D M. Barton *Vacuum* 3, Jan. 1953 51-53

Coupling Glass Tubing to Other Materials — 73/II

United States. In order to overcome the difficulties of joining glass tubing to piping or tubing of other materials such as steel or copper, a series of interchangeable parts have been designed from which a proper joint can be assembled without having to use improvised means. The joints have been tested for leaks at pressures up to 70 lb./inch². and under vacuum. No tools are required to tighten them. The joints can be used for the connecting of water jackets to condensers, and in cases where rubber joints are inadvisable and ground joints are undesirable because of the lubricant required. Details of the construction are given together with illustrations.

Sommaire : Détails concernant la construction et les dimensions de joints pour tubes en verre avec d'autre matériaux, resistant à des températures et pressions modérées.

Article by T. R. Tait & J. T. Mitchell *Ind. Engng. Chem.* 45, Jan. 1953 239-240

Vacuum Tightness of Welded and Brazed Aluminium Containers — 74/II

See Abstract No. : 94/I

Plating as an Aid in the Brazing of Stainless Steel — 75/II

See Abstract No. : 95/I

A Metal Vacuum Valve — 76/II

Holland. The body of the valve described carries three connections, one to the backing pump, one to the diffusion pump and one to the vessel to be evacuated. The latter two are in a vertical position. Their ends are covered by a rotating disc provided with an aperture which, in the appropriate positions of the disc, connects the rotary pump with the diffusion pump or the vessel to be pumped. In a third position, at about 90° to the previous settings, both connections are shut off and a leak-in valve opened by means of a ball situated in the periphery of the disc facilitating the admission of air to the backing pump. At the same time a microswitch is operated which switches off the backing pump. When the disc is returned to either of the two former positions the switch is again operated and the pump re-started. ' O '-ring seals are used throughout as sealing elements and have been found to be in good condition even after 5,000 revolutions of the disc.

Sommaire : Description d'une vanne à vide à trois voies.

Letter by G. M van Koppen *Appl. Sci. Res.* 3B, 1953 141

Improvements in Apparatus for Introducing Specimens into an Evacuated Chamber — 77/II

See Abstract No. : 58/II

A Vacuum Lock for Routine Solid Analyses With a Mass Spectrometer — 78/II

See Abstract No. : 59/II

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Contd.

Abstract No. and References

Apparatus for Supplying Charge Material to Mass Spectrometers 79/II

See Abstract No. : 60/II

Safety Trap for Vacuum Line 80/II

South Africa. Where vacuum is used to transfer liquids from one storage vessel to another a safety trap should be provided to prevent the liquid from being drawn into the vacuum system. The design of a trap is described and shown in an illustration which is employed in a system transferring distilled water from the still to an overhead distribution tank. In operation, any liquid overflow must pass into the trap before it reaches the vacuum system. The liquid collecting in the trap causes a float to rise, which operates a valve closing the connection to the vacuum system. Simultaneously a needle valve opens and admits air to the trap. Now, the trap can be drained by a stopcock. The trap is reset for normal operation by depressing the needle valve.

Sommaire : La construction d'un piège est decrit, lequel empêche un liquide de pénétrer dans un système sous vide. Il est utilisé, dans un système transferrant de l'eau distillée d'un distillateur à un réservoir.

Article by R. S. Young & R. Snaddon *Lab. Practice* **2**, Jan. 1953 27

29 — MISCELLANEOUS APPARATUS — 29

Bursting Disc Assembly for Alternating Pressure and Vacuum at Elevated Temperatures 81/II

See Abstract No. : 96/I

A Recording Mercurial Manometer for the Pressure Range 0-760 mm. of Mercury 82/II

See Abstract No. : 47/II

Environmental Test Chamber 83/II

United States. This is a short note describing a recently installed 440 ft.³ environmental test chamber for the testing of sub-assemblies and materials required in high-flying aircraft. The chamber can produce variable temperatures, humidity and altitude conditions, all of which may be changed independently of each other. In addition, the chamber is equipped with a 3,000 r.p.m. drive and other auxiliaries to simulate actual operating conditions if desired.

Sommaire : Brève note sur une chambre à atmosphère variable pour l'étude de produits finis, pièces détachees etc., pour avions.

Note by Anon. *Instruments* **26**, Feb. 1953 274

Metal Ebulliometers for Vapour Pressure Measurement 84/II

United States. There are many variations of the ebulliometric method of measuring vapour pressure but only a few are available for use with corrosive materials. The equipment described here has been developed for corrosive materials and can be used for the entire liquid range. Two apparatus are mentioned, the first, for the determination of vapour pressures up to two atmospheres and the second for the determination of vapour pressures as high as 3,000 psia. The first consists of a single boiling point tube where the sample is heated electrically to its boiling point at selected pressures. A platinum resistance thermometer was used for the measurement of the dew point temperatures of the vapour. Coiled copper tubing through which a stream of air could be passed is suspended in a vacuum jacket surrounding the boiling point tube as an additional means of controlling the temperature in the column. A condenser filled with ice water prevented the sample from entering the manometer system. This latter consisted of two small liquid nitrogen traps, a surge tank, a soda-lime trap, the controls and a mercury column manometer with a three-metre long reference limb made from 50 mm. precision bore tubing. The reference limb was precisely regulated by continuously bleeding helium to the vacuum pump. Through a different valve high pressure helium was admitted intermittently into the system to act as a buffer between the boiling point tube and the manometer. Details are given of the manustat arrangement incorporated in the system. Initial experiments with benzene yielded data deviating no more than 0.04° from those published by the National Bureau of Standards. The second apparatus consisted of two identical boiling-point tubes connected to a common pressure system. One boiling-point tube was filled with water and constituted a reference system. The vapour pressures of water are accurately known. Thus measuring the boiling temperature of each at the same pressure facilitated the determination of the pressure of the test sample. The temperatures were again measured with a platinum resistance thermometer. Both boiling-point tubes were connected to a common helium supply, surge tank and pressure gauge and a small valve used for evacuating the system releasing excess pressure, etc. The apparatus was used to determine the vapour pressure of n-hexade-cafluoroheptane in the range of 90–105°C. The values obtained by the two methods did not differ by more than 0.05% of the pressure.

Sommaire : Détails sur un dispositif de mesure de pression de vapeur fonctionnant en rapport avec la méthode ebulliometrique convenant à l'emploi de matérial corrosif.

Article by G. D. Oliver & J. W. Grisard *Rev. Sci. Instrum.* **24**, March 1953 204-206

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Abstract No. and References

30 — EVAPORATION — 30

Vacuum Deposition becomes a Trade 53/III

United Kingdom. The layout, general production programme and production methods of a company formed recently for the sole purpose of vacuum coating ' to the trade ' are described. The majority of articles received for treatment are made of plastics but a number of them are made of metal, in particular zinc-based die cast alloy. The most important feature of the production procedure is the method of jigging. The jigs are specially designed to suit the individual types of articles processed and after the initial loading, the articles remain on the jig throughout the whole sequence of operations. After degreasing, the parts are dip-coated with the base lacquer and stoved. Stoving temperature for metal parts is 110°C. but few plastic materials can be heated above 55°C. without harm. This makes it difficult to find a suitable lacquer for the latter. A.o. the base lacquer must withstand any possible attack by solvents held by the lacquer used for the top coat and likely to penetrate the pores of the metal layer. Good protection is obtained if the lacquer polymerises at the stoving temperature. Three 24 inch. vacuum chambers are available for the actual metallisation process. After metallisation the articles are dipped to receive the top coat. These lacquers may also be of the polymerising type and should be completely transparent, colourless and have good resistance to abrasion by handling. A total of six electric ovens are provided for the stoving operations. Comparing the vacuum process with the silver reduction method from the costing angle it is stated that the latter requires less capital equipment but higher running expenses than the former. Comparison with the electrolytic method is difficult as the two methods are normally applied for different purposes. Electrolytic deposition is to give a protective coating in addition to the decorative effect, whereas at the present time vacuum coating is essentially applied for decorative purposes only. However, the vacuum coating process appears to be the cheaper of the two methods. Its potential uses to industry are appreciable and should be more extensively exploited.

Sommaire : Description des méthodes de production et des installations d'une firme spécialisant dans la métallisation sous vide.

Article by Anon. *Prod. Finish.* Jan. 1953

High Quality Plating in a Vacuum 54/III

United States. The basic principle of the method of coating by evaporation in vacuum is explained. A shop layout for the metallisation of articles in batches is shown in a diagram giving the sequence of operations at the same time. The plan provides for one 48 inch. vacuum coating plant. Before operations begin the work pieces are clamped to riser rods and the rods conveyed by truck or belt to the various processing stations. It is suggested to use automatic dipping machines for the lacquering operations and drying tunnels for stoving. On arrival at the coating plant the riser rods are transferred to a station holding fixture which can accommodate up to 100 rods. Each station rotates around its own axis and several stations are linked by chain and sprocket drive to expose all sides of the work piece to the metal vapour beam. Compared with conventional methods the vacuum metallising process offers three major advantages, higher economy, reduced handling and greater safety in operation.

Sommaire : Revue des procédés de métallisation sous vide.

Article by Anon. *Mill & Factory* March 1953 96-7

The Application of Aluminium Reflecting Films to Glass and Plastics by Thermal Evaporation. Part II 55/III

Canada. The article presents a study of the fundamental principles which govern the deposition of aluminium films upon various materials. Part I published in the preceding issue Vol. II, No. 4 pages 365 to 376, dealt with aluminium films on glass (front surface mirrors). Part II, the present and final instalment, deals with conditions regarding the deposition of aluminium on plastics, and gives a very detailed account of the lacquering technique for under- and over-coating the aluminium films, of particular value to the commercial processor of decorative coatings.

Sommaire : Deuxième et dernière partie d'un article traitant des conditions concernant le dèpôt d'aluminium sur des plastiques (1er partie, voir Abstract No. 188/III, Vol. 2.)

Article by S. Bateson *Vacuum* 3, Jan. 1953 35-42a

Uniform Surface Metallisation by Evaporation From One or More Emission Surfaces 56/III

France. Describes the detailed design of an evaporation coating apparatus in which the surface to be metallised is rotated over the source.

Sommaire : Description de la construction spéciale d'un appareil à metallisation sous vide pour l'obtention de couches uniformes.

(*Science Abstracts*)

Article by P. Prugne & P. Leger *J. Physics Radium* 13 (Suppl. No. 7, 8, 9) July, August, Sept. 1952. 129A-130A

The Limit of Accuracy in the Measurement of the Thickness of a Thin Film Using the Koester Interference Prism 57/III

See Abstract No. : 104/I

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Abstract No. and References

The Formation of Adsorbed Layers in Vacuum Chambers and their Detection by Electrons 58/III

See Abstract No. : 77/I

Apparatus and Process for Coating a Substrate with a Metal 59/III

United States. Coating with aluminium by vacuum evaporation requires that the aluminium has a temperature of 1,200-1,600°C. At this temperature aluminium is highly reactive and care must be taken that the crucible is of a material inert to the molten aluminium, such as graphite and carbon. But graphite and carbon are readily wetted by aluminium and consequently the aluminium tends to climb over the side of the crucible. This effect causes wastage of the metal and may lead to the destruction of the furnace materials by the hot metal which has dripped over the crucible edge. To improve these conditions it is proposed to use a graphite crucible, cylindrical in shape with a lip at the top, which is placed in another cylindrical container of a larger diameter and made of silica, holding refractory material such as beryllia or zirconia. The crucible assembly is fitted to the base plate of the vacuum chamber. Heating is supplied by the induction method. The induction coil surrounding the crucible assembly has closer turns near the top than near the bottom of the crucible. It is suggested to use a power supply of about 100,000 500,000 c./s. At 100,000 c./s. the induced current will penetrate the graphite-carbon mixture known as ' Graphitar ' to a depth of 0.2 inch. Thus, if the walls of the crucible are slightly thinner than 0.2 inch. some of the induced current will reach the aluminium charge. In operation, the charge fills the crucible to within half an inch from the top and the molten aluminium creeps onto the lip as before but the temperature of the aluminium on the lip is about 100°C. higher than that in the crucible proper. As a result the aluminium evaporates before it reaches the outer edge of the lip. In this way a higher rate of evaporation and an increase of vaporising area is obtained. More than 4 grams per minute per square inch. of the surface area of the crucible body can be evaporated with this device at temperatures of the order of 1,350°C. and at pressures of about 0.2 micron Hg.

Sommaire : Description d'une source d'évaporation en forme de pot destinée à éliminer le débordement d'aluminium fondu et donner une surface d'évaporation étendue.

National Research Corporation *Brit. Pat.* 685,269

A Technique for Making Molybdenum Crucibles for Ceramic Research 60/III

See Abstract No. : 129/I

Improvements i.o.r.t. Metal Vaporisers 61/III

United Kingdom. The method described is applied in the production of incandescent filament lamps of the type which have a bulb with a flat frontpart to transmit the light and internally metal-coated side walls acting as a reflector. The metal-coating, normally a silver film, is applied by vacuum evaporation and the method employed must be suitable for mass production. The proposed design of the evaporation source consists (for lamps of usual size) of a 25 mil molybdenum wire, about 213 mm. long carrying a winding of 6 mil molybdenum wire (78.8 turns per inch). This coil-on-mandrel assembly is annealed and then shaped to form a convoluted centre portion and two legs. The latter are supported by the cover of the exhaust port of the pumping unit evacuating the bulb. One leg is in electrical contact with the pump housing, the other is insulated from it. After shaping the assembly is fired in a hydrogen atmosphere at about 1,500°C. and then plated electrolytically with silver. About 150 mg. of silver are required for one charge. After a final firing process the vaporiser is ready for use. During the evaporation process the convoluted portion of the vaporiser is in the focus of the lamp envelope and the latter is heated and pumped continuously. After completion of the deposition process the silver on the flat front part of the bulb is removed chemically. The method described here is claimed to represent an improvement of the procedure covered by British Patent No. 550,848 as it prevents the molten silver collecting in large globules which it is difficult to vaporise. Silver plating has proved to be superior to the use of silver wires as the charge. Also, molybdenum is not wetted by silver. Thus, the vaporiser remains free from contaminations and can be used repeatedly.

Sommaire : Description d'une méthode de depôt réflecteur d'argent à l'intérieur d'une lampe à incandescence de forme convenable, la couche métallisée servant de reflecteur à la lumière émise par la lampe.

The British Thomson-Houston Co. Ltd. *Brit. Pat.* 688,303

Method for the Deposition of a Thin Metal Layer on the Rough Surface of the Screen of a Cathode Ray Tube 62/III

Germany. The method is applicable in the production of cathode-ray tubes with fluorescent screens supported by a thin metallic layer. In many instances, the support is provided by materials with properties which ensure high-quality sealing with glass, such as nickel-iron and nickel-iron-chromium alloys. During the sealing process, the surfaces of these materials become dirty and rough. This prevents good adhesion and uniformity of the fluorescent layer, even in the case where a thin metallic intermediary layer of tungsten, molybdenum or aluminium is provided. It is proposed therefore to cover the rough surface first with a coating of an organic material such as collodion dissolved in amyl acetate which will produce a flat and uniform surface on the metal support, then to deposit the thin metallic film by vacuum evaporation onto which the fluorescent layer is applied. The organic intermediary layer is removed by evaporation during the baking of the valve.

Sommaire : Afin d'obtenir un support lisse pour la couche métallique formant la face arrière de l'écran fluorescent d'un tube à rayon cathodique un film intermédiaire est déposé sur le support métallique et enlevé par l'évaporation durant le récuit.

Telefunk Ges. F. drahtlose Telegraphie mbH. *Ger. Pat. Appl.* 02283

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Methods of Increasing the Electrical Conductivity of Surfaces — 63/III

See Abstract No. : 91/I

Mean Free Paths of Electrons in Evaporated Metal Films — 64/III

United States. The preparation of thin films of Cu and Ag was examined. Optimum condition was obtained by using high rates of evaporation (500-1,000 Å/sec.) and low residual gas pressure (0.00001mm.). Films evaporated at slower rates, at higher residual pressures, or deposited by chemical or electrolytic processes depart from calculated values. The resistivity and resistance temperature coefficients are reported at 25° for films of Cu and Ag 100-1,500 Å thick. From these data the mean free paths of the conduction electrons in these metals can be calculated.

(*Chemical Abstracts*)

Sommaire : Le mécanisme de la conductivité éléctrique de films minces a été étudié.

Article by F. W. Reynolds & G. R. Stilwell *Phys. Rev.* 88, 1952 418-419

Infra-Red Spectra of Evaporated Films — 65/III

See Abstract No. : 114/I

Mirror Coatings for Low Visible and High Infra-Red Reflectance — 66/III

See Abstract No. : 103/I

Apparatus for the Measurement of Reflecting Power in the Vacuum Ultra-Violet — 67/III

See Abstract No. : 101/I

Measurement of the Reflected Power of Thick Metallic Layers (Au, Pt, Cr) in the Schumann Range. Determination of Optical Constants — 68/III

See Abstract No. : 100/I

31 — CATHODIC SPUTTERING — 31

Methods of Increasing the Electrical Conductivity of Surfaces — 69/III

See Abstract No. : 91/I

Electrical Conductivity and Structure of Sputtered Cadmium Oxide Layers — 70/III

Germany. Cadmium oxide has been the subject of a number of investigations as it is known to have an unusually high electrical conductivity. Thin films of this material are of particular interest because they show good transparency for visible light. But the production of such films by the evaporation method has the drawback that the oxidation of the cadmium has to be carried out as a separate process after deposition. By using the sputtering method, however, cadmium oxide layers can be produced directly by the so-called process of 'reactive sputtering'. Cadmium oxide layers of good transparency varying thickness and reproducible conductivity values can be obtained by this method. A thorough study of the conditions of the process is reported in the present paper. The density of the films was found to be 7.6 g./cm.^{3} as distinct from 8.15 g./cm.^{3} published previously for the bulk material. The experiments were carried out in a sputtering apparatus which permitted operation in a continuously flowing gas. This ensured continuity of the chosen composition of the sputtering atmosphere. The conductivity of a cadmium oxide layer is independent of sputtering time, but the sputtering load and the nature of the sputtering atmosphere, in particular, the value of the partial oxygen pressure have a marked influence. The layers are binding a considerable amount of gas during the process of deposition. This could be proved by examinations in the electron diffraction camera and by dissolving the layer chemically in diluted sulphuric acid, collecting the gas evolved in the process. The studies proved that the gas does not only enter cavities in the layer but also enters the individual cadmium oxide lattice in atomic form. Oxygen and nitrogen can enter the lattice without difficulties but argon cannot because the diameter of the argon atom is too great. The incorporation of nitrogen in the lattice increases the conductivity appreciably but only those nitrogen atoms are effective which take up the position of an oxygen atom in the lattice. The conductivity of layers deposited in a pure oxygen atmosphere or in an argon-oxygen mixture is likely to be governed by the amount of excess cadmium included in the lattice. A reduction of the oxygen content of the sputtering atmosphere and an increase of the sputtering load should favour such inclusions. This was confirmed by experiment. The temperature coefficient and electrical performance of cadmium oxide layers heated in air varied depending on the method of their production. But at a temperature of 400°C. the lattice defects which seemed to have caused these variations disappeared and conductivity values measured were identical with the known values.

Sommaire : La conductivité électrique et la structure de films d'oxyde de cadmium appliqués par atomisation cathodique dans divers mélanges gaseux fait l'objet d'une recherche.

Article by G. Helwig *Z. Phys.* 132, 1952 621-642

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33 — IMPREGNATION — 33

Sorbed Gases and High Vacuum Systems 71/III

See Abstract No. : 79/I

34 — FUMIGATION — 34

Study of Vacuum Fumigation Techniques 72/III

United Kingdom. A brief progress report is given on fumigation experiments mentioned for the first time in the previous annual report (see Vol. II Abstract No. 197/III). The experiments were carried out in a chamber of about 1,700 litre volume at 15°C. room temperature and at 70% relative humidity. The fumigant was methyl bromide and the product a single bag of wheat feed. Evacuating the chamber first, introducing the fumigant and raising the pressure immediately afterwards was more effective than the atmospheric method. Maintaining a low pressure of 7 cm. Hg over a period after the fumigant had been introduced gave still better penetration into the bulk. The highest concentrations of the fumigant in the centre of the bag were obtained, when the vacuum was released early in ' sustained-vacuum ' tests. Breaking the vacuum after one hour in a 4-hour fumigation test appeared to give optimum results. It was observed that the fumigant was sorbed rapidly by the product during the first two hours of a test, the total amount sorbed being roughly proportional to the free space concentration.

Annual Report by Pest Infestation Res. Board *D.S.I.R.* 1952 31-32

Sommaire : Bref rapport sur les essais de fumigation, décrits pour la première fois sous résumé 197/III, Vol. II.

Behaviour of Fumigants During Vacuum Fumigation—I. Penetration of Methyl Bromide into Boxes of Dates 73/III

United Kingdom. The following vacuum fumigation methods are commonly used for the disinfestation of stored products : The sustained-vacuum method, the released-vacuum method and a special development of the released-vacuum method. In the case of the first, the chamber holding the product is evacuated to 5 cm. Hg and subsequently the fumigant is introduced. The low pressure is maintained for the whole of the fumigation period. In the case of the second, the chamber is evacuated, the fumigant introduced and the pressure raised to atmospheric immediately afterwards. In the case of the third, the chamber is evacuated and subsequently a mixture of air and vaporised fumigant is admitted continuously until the pressure in the chamber has reached atmospheric. Few data is available which facilitates an assessment of the comparative merits of vacuum methods and of vacuum methods as a whole with respect to atmospheric methods. The authors undertook a comparative study of the sustained-vacuum method the released-vacuum method and the atmospheric method. The fumigant was methyl bromide. The measured penetration of the fumigant into the product was used as the discriminating factor. A chamber of 1,700 l.capacity was used in the experiments situated in a room controlled at 16° and 70% relative humidity. In a few experiments the products fumigated were wheat and decorticated ground nuts in bags of 140 lb. each but normally the test product was compressed dates in 70 lb. wooden cases 19 × 11 × 10 inch. size. Details are given of the methods employed for the introduction of the fumigant and the withdrawal of gas samples for analytical purposes. The concentrations determined from the samples were plotted against time. The areas below these curves facilitated estimating the concentration-time products in mg. hr./l. for any given fumigation periods. The nominal concentration could be calculated from the quantity of fumigant used and the volume of the chamber. By expressing the measured concentration-time product as a percentage of the nominal concentration-time product, the efficiency

Table I. Summary of Results

Expt. No.	*Method*	*Products*	*Sampling Position*	*Nominal Concn., mg./l*	*Period hr. min.*	*Concn.-Time product, mg. hr./l.*	*Penetration Factor*
2	Atmospheric	Dates, 2 boxes	Centre, box 1	37	3 —	3	3
			" "		24 —	240	27
			Centre, box 2		3 —	11	10
			" "		24 —	290	33
3	French Released-Vacuum	Dates, 1 box	Centre	36	3 —	34	32
6	Sustained Vacuum*	" "	2 inch. deep	96	3 —	145	51
			" "		6 —	560	98
			Centre		3 —	110	38
			"		6 —	650	113

* Vacuum released after 3 hr. 30 min.

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Table II. Penetration Factors Obtained by Two Techniques at Centres of Boxes of Dates in Equal Periods of Time.

Expt. No.	*Load, boxes*	*Total period* Hr.	Min.	*Penetration factors* *sustained-vacuum only (estimated)*	*with delayed release of vacuum*
5	1	4	10	54	113
6	1	4	10	46	75
		6	—	55	113

of penetration of fumigant to the point sampled could be indicated by the so-called ' penetration factor ', as shown in Table I partly reproduced on the previous page. The concentrations obtained in the various experiments are given in a series of diagrams. Atmospheric tests on dates yielded penetration factors of 3 and 10 after 3 hours at the centre of the box. But the concentrations at these points rose continuously during the (normal) 24 hour processing cycle indicating that dates could be fumigated effectively at atmospheric pressure if dose and exposure time was adequate. In experiments with the released vacuum method initial diffusion to the centre of the block of dates was slow and for 30 minutes the concentration at this period was kept low due to sorption but subsequently it began to rise and the penetration factor after 3 hours was 32, almost the same as that obtained in the 24 hours cycle of the atmospheric method. Where the penetration factors obtained in a 3-hour process and a 24-hour process are the same, the dose of fumigant required for the 3-hour process is very much higher than that required for the 24-hour process. A single box of dates was tested by the sustained vacuum method. After 3 hours the concentration at the centre was about 73 and the penetration factor 45. It was observed in this experiment that the concentration at the centre increased appreciably when the pressure in the chamber was raised at the end of the treatment by allowing air to enter the free space. As this could have a marked effect on the efficiency of the process tests were carried out with a modified sustained-vacuum method providing for the continuation of the treatment for a limited period by admitting air at the end of the low pressure treatment without removing any of the fumigant present. The results of these experiments are shown in Table II, partly reproduced above. It is pointed out that the increase in the efficiency of penetration observed in the experiment would be less marked in commercial fumigation where the chamber is normally more tightly packed with the commodity to be fumigated.

Sommaire : Rapport detaillé sur une expérience fait pour le D S.I.R. sur l'éfficacité de différentes méthodes de fumigation sous vide.

Article by W. Burns Brown and S. G. Heuser *J. Sci. Food & Agric.* **4**, Jan. 1953 48-57

36 — DRYING — 36

Bibliography of Industrial Drying 74/III

See Abstract No. : 83/I

Dehydrated Meat 75/III

See Abstract No. : 30/IV

Puffed Powder from Juice 76/III

United States. Details are reported of the ' puff-drying ' process developed by The Western Regional Research Laboratory, U.S.A. for the production of powders from fruit and vegetable juices which will reconstitute almost instantaneously in water at all temperatures. The method provides for the use of a vacuum dryer operating at 29 inch. pressure and temperatures up to 300°F. The starting material is a rather viscous vacuum-concentrated juice containing at least 50% soluble solids. The material is fed into metal trays until the bottom is covered by a layer of limited depth. The trays are placed in the vacuum dryer on tubes or hollow shelves heated by steam or water. The temperatures are high during the initial stages but are gradually reduced as drying continues. During the process the material expands up to 20 times its original volume and assumes a sponge-like structure which assists the drying action. No excessively low pressures are required. Heat sensitive substances such as oranges can be dried to powder within one hour if the depth of the liquid in the trays does not exceed ¼ inch. When dry, the powder is removed from the trays, crushed, screened and packed at room temperature but in a dehumidified atmosphere. Flavours volatilised during processing are reincorporated in the powders. In order to prevent oxidation during storage the flavours can be ' locked ' in molten Sorbitol. The two-component emulsion is solidified, crushed, and admixed to the finished dried powder. To facilitate long storage life the powder must not contain more than 1% moisture. By providing in-package desiccation, however, it may contain as much as 3% moisture when packed in hermetically sealed containers along with a

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desiccant such as CaO which is placed in a moisture permeable but sift-proof bag to prevent contamination of the powder. Tests on orange powder have shown that moisture content had dropped from 3% to 0.5% during six months storage. The process has been applied with success to the production of powders from oranges, lemons, apples, tomatoes, pineapples, grapes and grapefruits, although in the case of tomatoes and other fruits giving a water-insoluble pulp a special technique is needed.

Sommaire : On donne des détails sur un procédé de dessiccation spécial employé dans la fabrication de végetaux et fruits en poudre à partir des jus.

Article by S. I. Strashun & W. F. Talburt *Food Engng.* **25**, March 1953 59-60

Improvements i.o.r.t. a Process and Apparatus for Evaporating Liquids

77/III

United States. A process for the evaporation and concentration of fruit juices or other aqueous solutions under vacuum is described where the heat liberated during the liquefaction of a compressed refrigerant, such as ammonia, is utilised for the evaporation of the water and the heat liberated by the water vapours during condensation is taken up by the cool refrigerant causing it to vaporise. In the particular plant described the ammonia is compressed to 205 lb. gauge pressure with a super-heat of up to 180°F. The liquid refrigerant leaving the evaporator has a temperature of 102°F. which is reduced to about 50°F. by special means during the passage of the refrigerant from the evaporator to the condenser. The vaporised refrigerant leaving the condenser is re-compressed to the above-named pressure before it is re-cycled through the evaporator. Vaporisation of the refrigerant in the condenser is facilitated by maintaining the compressor suction pressure at 75 lb. gauge pressure. The juice is treated under a vacuum of 28-29 inch. Hg. The vacuum is essentially created by the condensation of the vapours and is sustained with the aid of an ejector pump fitted to the condenser unit for the removal of non-condensable gases and 2 other pumps for the withdrawal of the concentrate and the condensate respectively. Evaporation takes place at about 60°F. A three-stage evaporator unit is used. All stages are of the same design, *i.e.* falling film evaporators which are operated in series. Means for re-circulating the juice are provided. The condenser is of the vertical, tubular type. Juice treated in accordance with this method can be concentrated from 11 Brix to 55 Brix density. In order to improve the flavour of the concentrate, fresh juice can be added after processing, diluting the final product to 44 Brix. It is advisable to heat-treat the juice to be processed in order to kill any constituents that might interfere with its preservation and to de-airate it to prevent foaming.

Sommaire : Description d'un procédé pour la concentration de jus de pamplemousse faisant usage de la chaleur latente libérée durant la liquéfaction du réfrigérant pour le chauffage et utilisant l'appel de chaleur pendant sa ré-evaporation pour la condensation du jus.

A. H. Stevens & Mojonnier Bros., Co. *Brit. Pat.* 686,375

Simple Laboratory Evaporator

78/III

United States. A modification is reported of the rotary film evaporator described by Partridge (See Abstract 113/III, Vol. I) used for the concentration of liquids. In the present design the whole driving shaft assembly and the bearing are made from glass. The bearing is fabricated from the plunger and housing of a standard Becton-Dickinson 20 ml. syringe, eliminating the need for special packing glands. The only other material used is rubber for stoppers. An ordinary laboratory stirrer can be employed as the drive. The vacuum obtainable depends on the fit in the bearing and the quality of lubrication (glycerol). Using a mechanical pump pressures from 5 to 25 mm. Hg can be maintained. The modified evaporator can be operated satisfactorily at 60 r.p.m.

Sommaire : Il est rendu compte d'une modification de l'évaporateur à film tournant décrit dans l'extrait No. 13/III, Vol. 1.

Note by P. Numerof & K. Keinhardt. *Analyt. Chem.* **25**, Feb. 1953 364

37 — METALLURGICAL PROCESSES — 37

Rapid Graphical Procedure for Oven Design

79/III

See Abstract No. : 80/I

Casting Under Extreme Conditions

80/III

Austria. Apparatus and technique employed in the following modifications of normal casting practice, and benefits derived therefore, are discussed : (1) Casting in vacuo; (2) solidification under pressure up to 20.000 atmospheres; (3) extremely slow and extremely rapid solidification and (4) crystallisation under the influence of ultrasonic vibrations. A plant for the fractional refining of raw zinc under reduced pressures is operating in New Jersey. The crude metal is distilled in a carborundum muffle, and in the first so-called lead column, pressure and consequently the temperature are so adjusted that practically all the zinc and cadmium is vaporised, while any impurities present, *e.g.* iron, copper, tin, antimony and a little zinc remain at the base of the column. In the second column, termed the cadmium column, condensed zinc from the first column (together with its cadmium content) is subject to such a temperature that the cadmium evaporates and very pure zinc is retained. The solution of iron in zinc can be considerably speeded up by ultrasonic vibrations, and the same agency can be used to effect grain-refining by fracture of crystallites.

(*Z.D.A. Abstracts*)

Sommaire : Les effets de la pression et la variation de la vitesse de solidification sont décrits, ceux de vibration ultrasonique sur les propriétés de fontes sont discutés particulièrement.

Article by E. Schmid *Metall* **6**, Dec. 1952 737-744

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Titanium : A Survey

81/III

United Kingdom. Titanium, although the seventh most abundant metallic constituent of the earth's crust, is still a rare metal. The advantages it offers regarding density, corrosion resistance and mechanical properties are offset by the high cost of production. Now, 150 years after its discovery by Gregor, only two practical processes have been evolved for the production of titanium in ductile form, the Van Arkel and the Kroll process. The Van Arkel process yields the purer metal but is restricted by cost to application on a laboratory scale. The other, the Kroll process, simple in conception but involving complex chemical engineering problems, has virtually produced all of the titanium used to date. The process consists of reducing titanium tetrachloride with magnesium according to the equation $TiCl_4 + 2Mg = Ti + 2MgCl_2$. In practice the process yields 1 lb. of titanium sponge for $1\frac{1}{4}$ lb. of magnesium. The titanium tetrachloride is obtained from ilmenite concentrates containing 45% titanium dioxide by the reaction : $TiO_2 + 2C + 2Cl_2 = TiCl_4 + 2CO$. To avoid contamination, the decomposition of the tetrachloride shown in the above equation must be carried out in vacuum or in an inert atmosphere. The Kroll process was originally developed for batch production, the reaction chamber being completely welded up at the start and cut open at the end of the reaction. The conglomerate of titanium, magnesium chloride and surplus magnesium was bored out as chippings and chemically treated to remove magnesium products. The powdered titanium obtained by this method was compacted by powder metallurgy techniques. An important simplification was made to the process by applying vacuum distillation methods for the removal of the magnesium products. The sponge produced is arc- or induction-melted into ingots. Further improvements aiming at a continuous Kroll process may be expected to provide for stoichiometric quantities of molten magnesium and liquid titanium tetrachloride to be fed continuously into a reaction chamber, and the products to be discharged into an arc furnace where the titanium is melted into ingots and the magnesium products distilled and removed for magnesium recovery. This scheme has in fact been investigated in the U.S.A. with promising results. The following table gives the approximate cost of metallic titanium per ton in various forms based on current American prices and currency exchange rates. Its use as a structural material

Material	*Cost in Pounds Sterling of 1 Ton of Titanium*
	£
Ilmenite concentrate (45 per cent. TiO_2)	37
Titanium tetrachloride	500-600
Titanium sponge	4,000
Bars, hot-rolled	7,900
Forgings, rounds, discs, etc.	7,900
Sheet and strip	11,900

in civil aircraft will depend on the buyer. Its use in military aircraft will largely depend on the effectiveness of titanium as armour which has still to be tested. Its metallurgical potentialities are governed by its alloying properties. Binary alloys with metals of Groups 1B and V-VIII produce an increase in ultimate tensile strength but, with the exception of silver, a decrease in elongation. Certain ternary and higher alloys give greater static strength and less serious reduction in ductility. Alloys are formed with certain gaseous elements. Information on alloys with non-metallic solids is limited. Forging of the available titanium alloys is complicated and costly. Titanium and titanium alloys have been found to pick up on any other material, on which they are rubbing. This is a serious defect which may be overcome by appropriate treatment of the surface. Investigations on fatigue, impact resistance, welding and high temperature properties are continuing together with creep observations which show promise for some alloys.

Sommaire : Revue des méthodes de production de Ti.

Article by P. L. Teed *Engineer* 195, 6.2.53 203-205 & 195, 13.2.53 235-237

Improvements i.o.r.t. the Manufacture of and Coating by Titanium or Alloys of Titanium

82/III

Germany. A process of manufacturing titanium alloys is described whereby a titanium halide is reduced by an alkaline-earth metal, the latter being maintained in the solid state during the reaction. For example, the reducing metal, say magnesium, in the form of chips, grains or powder is reacted with the titanium halide, say tetrachloride, in vacuum or in a protective atmosphere. The magnesium is heated to a temperature of 500 to 550°C. and the tetrachloride added. The magnesium ignites and reduces the tetrachloride to titanium metal which combines with part of the magnesium to magnesium titanide, magnesium chloride being formed as a by-product. The reaction tube is placed in a sloping position to allow the magnesium chloride to run off. The magnesium is maintained below its melting point by regulating the amount of tetrachloride added. The titanide formed may be decomposed at a certain temperature and the magnesium distilled off. For producing titanium alloys other than alkaline-earth metal titanium alloys the titanide in small pieces, is mixed with another metal, having a higher boiling point than the alkaline-earth metal used; the mixture is heated and the alkaline-earth metal evaporated in a protective atmosphere. Also, an alloy may be formed by reducing a titanium halide together with a halide of the other metal or metals, with an alkaline-earth metal having a lower boiling point than the alloying metals. On completion of the reaction the alkaline-earth is distilled off. The principle of the method described can be used also for the coating of metallic or ceramic articles with titanium or titanium alloys.

Sommaire : Description d'une méthode pour la manufacture d'alliages de titanium comprenant la réduction d'un halogéné du titanium par un métal alcalino-terreux.

Titangesellschaft mbH *Brit. Pat.* 686,845

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Abstract No. and References

Improvements i.o.r.t. the Production of Coatings on Ferrous Articles 83/III

See Abstract No. : 123 I

Forging of Arc-Melted Chromium 84/III

United States. Chromium of high purity has great potential uses as a refractory and construction material for high-temperature engines if it can be worked by normal means. The lack of ductility of high grade electrolytic chromium is ascribed to its content of oxygen. The U.S. Bureau of Mines has developed a method of producing a metal of improved ductility by reducing it with hydrogen. After a short heat treatment at 1,000°C. in high vacuum electrolytic chromium contains about 1% Cr_2O_3. In order to remove this amount the chromium is powderised (60 mesh), leached, rinsed with distilled water and vacuum dried. The material is now ready for the reduction process and loaded in molybdenum boats into the furnace. The latter consists of 2 inch. diameter porcelain tubes which are part of a gas circulating system, to which hydrogen is admitted through a palladium catalyst tower to remove the oxygen in the hydrogen. Other major components of the system are :—A vacuum pump to exhaust the system, a small diaphragm pump to circulate the hydrogen, a getter furnace for the initial purification of the hydrogen filled with a zirconium-titanium alloy, a cold trap operated with a dry ice-acetone mixture and phosphorous pentoxide traps. During operations the reduction furnace is heated to a fixed temperature between 1 000 and 1,200°C. and the getter furnace to 890°C. The treatment lasts 16 hours. Subsequently the material, in lumps of a $\frac{1}{4}$–$\frac{1}{8}$ inch. size, was introduced through a vibrating feeder into an arc furnace details of which are shown in a sketch, and melted in a d.c. arc of 40 V and 800 A in an inert gas atmosphere (helium-argon of 80 : 20%). Approximately 750 g. chromium could be processed in 20 minutes. Metal treated in this manner contained Fe–0.001%; Si–0.05%; O_2–0.003% N_2-0.002% and H_2–0.005%. Tungsten pick up does not exceed 0.01. The hardness is R_b = 55. Annealing for one hour at 1,200°C. reduces the hardness to R_b = 46. The material is forged initially at 800°C. to break up the cast structure. Subsequently heavy reductions can be obtained, forging at 500°C. After forging the material is finally annealed in hydrogen at 1,200°C. The finished product shows ductility at temperatures considerably higher than described previously but it will fracture when bent at room temperature; this may be an inherent property of the material.

Article by Gilbert, R. Johansen, H.A. Nelson, R. G. *J. Metals* 5, Jan. 1953 63-65

Sommaire : Le Bureau des Mines Americain a developpé une méthode pour la production de Cr de ductilité raisonnable à des températures plus basses que celles connues jusqu'à present.

Determination of Hydrogen in Magnesium, Lithium and Magnesium-Lithium Alloys 85/III

United States. In connection with certain experiments the hydrogen content of magnesium, lithium and similar light metals had to be determined. The known methods of extracting the hydrogen gave erratic results. In order to overcome this, three special methods were tried, warm extraction at 500°C., extraction at 650°–800°C. from a specimen contained in a steel capsule, and tin fusion at 450°C. The first method was found to be impractical as the magnesium and lithium vapours reacted with the water adsorbed on the walls of the furnace. In the case of the second method, the sample was prepared in a dry-box, under an argon atmosphere. The steel capsule was weighed before loading and was closed with a steel plug and re-weighed after loading. It was then transferred to a chamber filled with argon for final welding. Then, the capsule was placed in a quartz vacuum tube furnace, heated to 800°C. and the hydrogen in the extracted gas determined by an Orsat apparatus. The procedure of determining the hydrogen content took about 11 hours in the case of a magnesium–16% lithium alloy sample of 5.24 g. The results are given in tabular form. The third method was carried out in a borosilicate glass (tube) furnace. Details of the arrangement are given in a sketch. 100 g. of tin were placed in the furnace tube. The sample was introduced into a side arm of the furnace. Then the furnace was sealed and evacuated. The tin was melted and degassed at 450°C. Subsequently the sample was pushed into the bath with the help of a pusher rod operated magnetically. When the gas evolution had subsided to about 0.01 ml. min. 15 g. of C.P. tin was dropped into the bath to ensure complete solution of the sample. About 1 hour was needed for each determination. In order to introduce pure lithium samples without causing a violent

Hydrogen in Magnesium Lithium, and Magnesium-Lithium Alloys
(Determined by the Tin-Fusion method)

Run No.	*Sample Weight Grams*	*Hydrogen* Weight %*	*Sample*	*Remarks*
7	3.98	0.0012	Commercial magnesium rod	—
26	2.36	0.0052	Mg-16% Li alloy 2663	—
11	0.645	0 065	Lithium 17629	Sample loaded in tin-foil capsule inside argon dry box
10	3.30	0.0039	Mg-5.3% Li alloy 2694	Edge of casting
50	3.22	0.0050	Mg-5.3% Li alloy 2694	Core of casting

* Precision of run 7 is ± 0.0003% hydrogen. Precision of all other runs is ± 10% of reported value.

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reaction, the bath temperature had to be reduced to 300°C. Once the sample had been added the temperature could be raised safely to 450°C. The results obtained by this method are given in a table partly reproduced on the previous page. Of the two successful methods the tin fusion method proved the more rapid and accurate. The steel capsule method was unsuitable for lithium only, due to permeation of the capsule walls. No pure magnesium samples were analysed by the capsule method.

Sommaire : Description de deux méthodes ayants permis de déterminer l'hydrogène dans du magnesium et des alliages Mg-Li.

Article by W. Mallett A. F. Gerds & C. B. Griffith *Analyt. Chem.* **25**, Jan. 1953 116-119

38 — DISTILLATION — 38

Improvement in the Functioning of Continuous Vacuum Distillation or Rectification Apparatus

86/III

France. An improved method of distilling and rectifying alcohol under vacuum is described. In apparatus of this kind a rotary vacuum pump is connected to the condenser and the air drawn into the pump contains some 5% (by weight) of the total quantity of alcohol obtained in the process. As the alcohol passing through the pump tends to dissolve in the pump oil, it is suggested to use liquid ring pumps, the bearings of which are the only parts requiring lubrication. In this case the water forming a liquid ring between the rotor and the body absorbs the alcohol drawn into the pump. In order to recover the alcohol the exhaust part of the pump is connected to a washing column operated with fresh water. A baffle in the washing column gives the incoming gas-water mixture a gyratory movement which causes the separation of the water from the gases. The latter rise in the column and bubble through sheets of water flowing in a downward direction through the column. On their way to a special outlet they leave the last traces of alcohol in the washing water. The water circulating through the washing column is collected in a tank fitted with cooling tubes where it is mixed with a certain amount of water coming from the liquid ring pump direct. After cooling, the water is returned from the tank to the liquid ring pump thus closing the water circuit of the pump. The water circulating through this closed circuit is gradually becoming richer in alcoholic content. In order to stabilise the alcohol content of the water in the circuit a certain amount of liquid is extracted continuously through a valve fitted with a suitable measuring device. The amount extracted is fed to the still. To make up for this loss fresh water is added through the washing column.

Sommaire : Méthode pour recouvrir l'alcohol entrainé dans l'air ou le gaz passant par le condenser d'un distillateur d'alcohol.

Société des Etablissements Barbet *Brit. Pat.* 683,781

Falling-Film Type of Vacuum Still with Rotating Partial Condenser

87/III

United States. A distillation apparatus is described which facilitates precision fractionation and is recommended for the processing of lubricating oils and similar materials. The apparatus consists of a pot holding the material to be processed which can be heated. The pot carries the fractionating column consisting of three coaxial cylinders and a central shaft. A pump submerged in the liquid in the pot passes the liquid material to the outermost annular space of the column which reaches to about half the full height of the column. From here the liquid passes through two heat exchangers where it can be heated or cooled as required before it is fed at the top of the column into the innermost annular space immediately surrounding the central shaft. This assembly (the wall and the shaft) is rotating and as a result of this the liquid now cooled to a pre-determined temperature flows downwards in the form of a film on the wall until it emerges at the bottom of the column in the form of a fine spray to rejoin the liquid in the pot. The central annular space accessible directly to vapours rising from the pot is thus formed by a hot, stationary outer wall and a cold, rotating inner wall. As a result the rising vapours are frequently condensed and re-evaporated before they reach the outlet of the column leading to the condenser. The necessary low pressure is provided by a vacuum pump fitted to the condenser.

Sommaire : Description d'une colonne de fractionnement pour le traitement de mélanges d'huiles à gros poids moléculaire, dans laquelle une partie des matières traitees sont utilisées pour chauffer et refroidir la matière sous traitement.

A. W. Goldsbarry M. S. Agruss The Pure Oil Co. *U.S. Pat.* 2,613,176

Distillation Apparatus for Refining Fats and Oils

88/III

United States. A method is described for the refining and deodorising of animal and vegetable oils which facilitates continuous operation. The plant consists of a large vertical stainless steel tube closed airtight at both ends. This tube carries a second tube of a smaller diameter, inserted from the top, the outer surface of which serves as a condenser face. At the bottom of the inner tube is a catch basin which can be drained to the outside by means of a suction pump. The space between the two tubes is evacuated through an outlet at the bottom of the large tube. The material to be processed is introduced near the top of the outer tube through a ring pipe, enveloping the inner tube, with holes in the downward direction. Immediately below the feeding ring is a second outlet in the outer container also connected to a vacuum pump. The large tube is fitted with two heating jackets separated from each other and situated at appropriate heights which can be heated with steam or any other suitable medium. The material entering the plant moves downwards by means of gravity along the inner

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wall of the outer tube. As it traverses the area heated by the upper heating jacket the more volatile adulterants are liberated. As it reaches the area covered by the lower heating jacket the less easily liberated adulterants are removed. These vapours move upwards through the annular space between the inner tube and the outer tube until they are withdrawn through the upper outlet. The interior of the inner tube is filled with a refrigerant. Consequently the condensable gases impinging on it are liquified and move downwards along the surface of the inner tube until they are collected in the catch basin and withdrawn to the outside. When the film of fat moving along the wall of the outer tube has reached the bottom of the evacuated chamber all impurities have been discharged and the material can be withdrawn through the bottom pipe communicating with the vacuum pump.

J. E. Thompson *U.S. Pat.* 2,611,741

Sommaire : Detail d'un désoderisateur développé récemment pour le traitement d'huiles animales et végétales·

39 — MISCELLANEOUS PROCESSES — 39

Thermal Conductivity of Glass Wool at Low Temperature Under Low Pressure of Air — 89/III

○ See Abstract No. : 107/I

The Micro-Analysis of the Inert Gases — 90/III

See Abstract No. : 111/I

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Special Equipment for Carbon^{14} Work — 91/III

United States. Two pieces of equipment have been designed for use where large amounts of carbon–14 are handled. One is designed for giving protection to an operator processing the compounds in a glass vacuum system. It consists of a wooden box 5 ft. × 4 ft. × 2 ft. deep, mounted on a base frame fitted with castors. The front doors are removed during the construction of the apparatus. Gas, water and vacuum are led in through outlets in the side of the box. All switches are mounted on a control panel externally, and may be isolated by a master switch. A removeable plastic lacquered steel tray, constitutes the bottom of the box. In operation, the box is closed except for small slits left open to allow for the manipulation of the apparatus. Details are given of the filter arrangements. The other piece of equipment consists of a hood mounted over a specially constructed glassware cleaning sink. The hood system draws air across the opening of the sink at a rate of 1,150 cu. ft./min. This facilitates the handling of noxious chemicals without hazard.

Article by B. M. Tolbert N. Garden & P. T. Adams *Nucleonics* 11, March 1953 56-58

Sommaire : L'on décrit deux pièces d'équipment, l'un protégeant l'opérateur durant la manipulation de larges quantités de carbone14 et l'autre facilitant la décontamination en sécurité.

Puffed Powder from Juice — 92/III

See Abstract No. : 76/III

○ **Deairating and Deoiling Citrus Juices** — 93/III

United States. A method is described for the removal of gases and oils from fruit juices, etc. which originate from the peel and may have passed into the juice during the pressing operation. No loss of juice occurs during the treatment. The apparatus consists of a steam-operated evaporator fitted with vertical tubes through which the juice rises. The top of the evaporator is connected to a vertical flash chamber. A circulation pump at the bottom of the flash chamber returns the juice to the lower end of the evaporator. Two valve-controlled outlets are provided in the pipe section between pump and evaporator, one for the admission of fresh juice and one for the withdrawal of finished processed juice. During treatment the juice is circulated through this system as long as required. A baffle in a suitable position in the flash chamber converts the stream of juice entering the chamber into a fine spray. This facilitates the separation of air, gases, vapours and oils from the body of the juice. Through the action of an ejector pump these components are drawn into a condenser. Here the non-condensable gases are removed to atmosphere by the pump and the condensate is collected in a vessel serving as an oil separator. Operating at a temperature of 125°F. and a vacuum of 26 inch. in the flash chamber and raising the temperature of the juice by 6°F. only during its passage through the evaporator, the oil content of citrus fruit juice can be reduced from 0.03% to less than 0.01% in conditions where government specifications calls for a maximum permissible of 0.015%.

M. J. Kerner & Blaw-Knox Co. *U.S. Pat.* 2,631,103

Sommaire : Machine pour opération continue construite pour éliminer des gaz et huiles de jus de fruits.

Distillation Apparatus for Refining Fats and Oils — 94/III

See Abstract No. : 88/III

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Powdered Mashed Potatoes

95/III

Sweden. Potato powder is now available manufactured by a vacuum process. The potatoes are peeled, boiled and subsequently the moisture content is removed by evaporation. The albumin, mineral salts and fats held by the potatoes are not affected by the treatment. One pound of potato powder is equivalent to twenty six pounds of whole potatoes. The consequent reduction in shipping space required makes the product cheap. Potato powder can be stored for four years without deterioration as compared with whole potatoes which will keep in good condition for only about nine months.

Sommaire : Note sur la préparation sous vide de poudres de pommes-de-terre étant récemment apparu sur le marché.

Note by Anon. *Compr. Air Mag.* March 1953

Improvements i.o.r.t. Metal Cans Sealed in an Airtight Manner

96/III

Holland. Normal practice in the canned food industry provides for filling the can, securing the lid to the can by means of interleaving flanges and soldering the seam by conventional techniques using a solder wire. While a satisfactory airtight seal is effected in this manner it has been found that the content of the can such as meat shows undesirable discolouration near the sealed end of the can, after opening. This can be avoided by using high-frequency heating in the soldering process, as heating by that method is more rapid with the attendant result that heat transfer by conduction to the contents of the can is reduced. Additional improvements can be obtained by designing the flanges in such a manner that soldering is effected in an area remote from the contents of the can.

Sommaire : Description d'une méthode de scellement de boîtes de conserves dans laquelle la téchnique normale de soudure a été remplacée par le chauffage à haute fréquence.

N. V. Philips' Gloeilampenfabrieken *Brit. Pat.* 685,597

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IV — Special Subsidiary Subjects — IV

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40 — GASES AND VAPOURS — 40

The Motion of a Gas Cloud Expanding into a Vacuum 19/IV

Abstract No. : 98/I

The Micro-Analysis of the Inert Gases 20/IV

Abstract No. : 111/I

Analysis of Synthesis Gas. Rapid and Highly Accurate Method 21/IV

United States. A rapid and accurate method for the analysis of synthesis gas is described which was developed for use in experiments on the kinetics of the carbon-steam and carbon-oxygen reaction. The synthesis gas to be analysed consisted of hydrogen, carbon dioxide, carbon monoxide and methane. The concentrations of the latter three never exceeded a total of 2% of the whole mixture. Normal Orsat procedures proved too slow. A mass spectrometer was not available. Finally, a gravimetric method was chosen based on the combustion and absorption of combustion products. The measurement of carbon dioxide presented no difficulty. The combustion of carbon monoxide, hydrogen and methane was carried out over platinum dispersed on silica gel and followed closely the procedure published by Kobe *et al.* Details of the gas combustion train are shown in an illustration. The apparatus was operated at atmospheric pressure. In order to check the performance of the apparatus gas samples of precisely known composition were required. These were prepared in a specially designed blending unit, which is described and shown in a sketch. It consisted of two parts, the gas storage and entry system, and the blending and mixing system. The essential components of the former were : A gas manifold to which bombs holding the various gases were attached, an open-arm manometer, a two-litre round bottomed flask, a Nesbitt bulb holding Anhydrone to dry the gas, a cold trap and a vacuum pump. The blending system contained a spherical vessel housing an impeller unit driven magnetically, an open-arm manometer with a levelling bulb and a two-litre mixing cylinder, the whole aggregate being installed in an air-conditioned room. A Toepler pump was incorporated in the blending system. In operation, the apparatus was first evacuated at the same time allowing the mercury to rise in the Toepler pump until it was level with the check valves. Then, the blending system was isolated and the heaviest component of the gas mixture to be prepared was admitted to the storage system. The amount of gas admitted was introduced into the blending system after the mercury in the suction leg of the manometer controlling the blending system had been raised to 900 mm. When the gas entering the blending section had depressed the mercury in the suction leg of the manometer to a pre-determined value, the blending system was isolated and the mercury in the manometer raised to the 900 mm. level again. The new level in the opposite arm of the manometer was a measure of the partial pressure of the first gas component admitted. Subsequently, the gas storage system was re-evacuated and the second gas component admitted in the same manner as the first. This was continued until all components making up the mixture had been admitted. After the last entry the impeller was started and the gases circulating through the unit were thoroughly mixed for 1 hour. This operation completed, samples of the mixture were withdrawn for analysis by means of the Toepler pump. The Table partly reproduced below, shows ' true ' and ' measured ' values of the four components in a number of samples prepared in the unit.

Analyses of Synthetic Samples

Sample No.	*Hydrogen, %*			*Methane, %*			*Carbon Dioxide, %*			*Carbon Monoxide, %*		
	Actual	*Determined*	*Error*	*Actual*	*Determined*	*Error*	*Actual*	*Determined*	*Error*	*Actual*	*Determined*	*Error*
1	71.82	71.74	0.08	3.19	2.92	0.27	14.77	14.70	0.07	10.22	10.64	0.42
4	35.44	35.40	0.04	18.03	18.33	0.30	28.18	28.09	0.09	18.35	18.26	0.09
8	85.59	85.56	0.03	3.46	3.35	0.11	4.75	4.87	0.12	6.20	6.22	0.02
10	29.40	29.44	0.04	22.76	22.93	0.17	24.34	24.12	0.22	23.50	23.51	0.01

Sommaire : On décrit une méthode pour l'analyse rapide de certains mélanges de gaz comprenant la description d'un mélangeur qui pourrait avoir une plus grande application.

Article by G. L. Barthauer, A. Haggerty & R. J. Friedrich *Analyt. Chem.* 25, Feb. 1953 256-259

Preparation of a Tritiated Self-Luminescent Material 22/IV

Abstract No. : 112/I

The Recovery and Purification of Tritium and He^3 Used in Ion Accelerators 23/IV

Canada. A method of recovering and purifying tritium and He^3 after their use as bombarding particles in ion accelerators is described. It was developed for the treatment of tritium and helium^3 withdrawn from the ion source of a specially designed 200 kV H.T. set, but is equally suitable for operation in conjunction with

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other accelerators. To reduce the risk of accidental losses the apparatus is made from metal and is fitted with O '-ring type valves and seals. Mercury pumps are used to avoid the possibility of an exchange between tritium and hydrogen in the hot oil. The gas withdrawn from the ion source is passed to a three-litre container fitted with a manually operated piston and communicating with a recovery reservoir. In the case of tritium the reservoir contains about 100 g. of spongy uranium which readily absorbs the tritium in the early stages but after a few hours, requires heating to about 200°C. to promote absorption and ensure that the residual pressure of gaseous tritium present in the container is kept below 1mm. Hg. A detailed sketch of the reservoir and the heating arrangements is given. When a fresh supply of tritium is required in the accelerator, the reservoir is connected to a gas supply bottle which feeds the ion source *via* a variable leak. The tritium is transferred to the bottle simply by heating the reservoir to about 450°C. At this temperature the tritium, stored in the form of uranium hydride, re-evolves as a pure gas. The evolution and supply of tritium is stopped by switching off the heat. The same apparatus is used for the recovery and storage of helium[3] but the storage reservoir has a volume of 1½ l. and is made of aluminium. Also, before the gas is used again, it has to be circulated by means of a Toepler pump through a special purification system containing a charcoal trap. The storage volume is 4½ l. As the pumps of the recovery unit can pump gas into that volume up to a pressure of 15mm. the storage capacity of this unit for helium at NTP is 90 c.c.

Note by K. W. Allen & E Almqvist *Rev. Sci. Instrum.* **24**, Jan. 1953 70-71

Sommaire : Détails concernant la méthode et l'appareillage utilisés pour récuperer et purifier le tritium et l'helium[3] après qu'ils aient été utilisés dans les accelérateurs à ions pour les bombardements.

A New Electron Multiplication Process 24/IV

Abstract No. : 90/I

Electric Discharges 25/IV

Abstract No. : 89/I

41 — FREEZE DRYING — 41

Bibliography of Industrial Drying 26/IV

Abstract No. : 83/I

A Method of Freeze Drying for Electron Microscopy 27/IV

Abstract No. : 55/II

Simple and Efficient Freezing Drying Apparatus for the Preparation of Embedded Tissue 28/IV

Sweden. The application of freeze drying methods for the preparation of embedded tissue has been suggested frequently in the past, see the publications of Packer and Scott, Taylor, Gersh, and Wang and Grossman, but the equipment proposed was rather complex. The authors developed a simple tissue holder and freeze drying apparatus which should facilitate the widespread use of this technique for the purpose. The tissue holder is a stainless steel rod with a handle at one end which carries 16 rings in 4 units of 4 rings each, screwed tightly to the holder. Each ring carries a sample of tissue not exceeding 2mm. in thickness on gauze tied to the circumference of the ring. After loading the holder is submerged in isopentane kept in a stainless steel container of about 5 cm. diameter which rests on, and dips into, the mouth of a Dewar flask filled with liquid nitrogen. The Dewar flask has a diameter of 6.8 cm. and a height of 15 cm. Normally the tissue is frozen before the *iso*–pentane has solidified completely. After about 30 seconds the tissue holder is quickly transferred to the freeze drying apparatus. Two designs, both of Pyrex glass are described and shown in illustrations. The second which represents an improvement over the first, consists of a sample tube connected by a ground glass joint to the exhaust tubulation. A freezing trap incorporated in the latter and operated with liquid nitrogen was only a few centimetres away from the sample tube after assembly ensuring short dehydration times in operation. An oil diffusion pump with a capacity of 50 l./sec. at a pressure of 1×10^{-5} mm. Hg was provided. The pressure was measured with a Philips gauge. Before the freezing trap was filled and prior to the dehydration of the tissue the sample tube was filled with the paraffin embedding medium (' Tissue mat ') to a depth of about 4 cm., joined to the exhaust pipe and the fore pump switched on. The paraffin was melted by surrounding the tube with a beaker of hot water. When the gas evolution from the molten paraffin had ceased the material was solidified by substituting a Dewar flask containing a solid carbon dioxide–organic liquid mixture for the beaker of hot water. The fore pump was turned off, the sample tube disconnected and the holder with the frozen tissue placed on top of the solid paraffin. The tube was reconnected, immersed in a

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Dewar flask with solid carbon dioxide and organic liquid and the dehydration process was started by switching on the high vacuum pump. The processing time varied in accordance with the mass of the specimens and the nature of the tissue. Four pieces of dog gastric mucosa (2 mm. cubes) processed at –80°C. and 10^{-5} mm. Hg were completely dehydrated in 6 hours. When dehydration was completed the Dewar flask was removed and the paraffin melted as before. As a result the tissue holder sank into the liquid. Bubbles rising to the surface during this operation indicated the degree of dehydration obtained. Where this continued for longer than a few minutes, the tissue was rejected. The tissue was kept submerged for about 5 minutes. Then pumping was stopped, the material cooled and the vacuum broken. The block of paraffin was removed from the sample tube and the excess paraffin trimmed away. The individual specimens were separated from the rings with the help of a dowel. The paper concludes with a discussion of suitable freezing temperatures and mixtures.

Article by D. Glick & B. G. Malmstrom *Exp. Cell Res.* 3, 1952, No. 1 125-135

Sommaire : On donne des détails sur une méthode, developpé recemment pour la lyophilisation de tissu.

Studies on the Stability of Lyophilised BCG Vaccine

29/IV

United States. The freeze drying method applied to the preservation of BCG vaccine, originally introduced as a means of combating the rapid loss in allergenic potency of the liquid vaccine, has shown up certain deficiencies, one of the most important being the apparent lack of protective value of freeze dried vaccines against challenge infection with violent tubercle bacilli. This could be attributed to high mortality of viable cells caused by the process. In preliminary experiments with the method the authors obtained low survival rates and in addition some proof that BCG cannot be successfully stabilised in the absence of drying excipients. The experiments were extended, therefore, by investigating a.o. the potential merits of 62 adjuvants belonging to the general classes of carbohydrates, organic acids, amino acids and their salts, proteins and digests. Nine strains of BCG and one strain of the vole bacillus have been used in the experiments : Strain 31, 186 425, 805, 826, 858, 862, 873, B59 and Strain 170V. On receipt a transfer was made to a slant of Lovenstein-Jensen's medium and the culture incubated at 38°C. For use the stock cultures were transferred to glycerinated water potato medium. For the preparation of the vaccine small portions of the veil of growth on the liquid phase of the culture were transferred to Sauton's medium, incubated at 38°C and the growth harvested after 10 days. The bacilli were separated from excess fluid and ground in a rotating flask. During this operation 50% glucose solution was added to bring the bacterial concentration to 0.2 mg. dry weight per microlitre of grinding menstruum. Then the suspension was centrifuged at high speed, the supernatant fluid removed and the cells were washed with M/15 phosphate buffer at pH7.0. Subsequently they were re-suspended in phosphate buffer to 1/10th of the original volume and finally adjuvant solution was added bringing the bacterial concentration to 1 mg. dry weight per ml. suspension fluid. Details of the standardisation of the vaccine suspension are given. The ampoules containing the test material were pre-frozen in a liquid bath of dry ice and acetone. Two freeze drying apparatus were employed, accommodating 62 and 46 ampoules respectively. A mechanical rotary pump was used for exhaustion. The operational pressure was 0.015 mm. Hg. The water vapour was removed by condensation, and the heat required was supplied by convection. The moisture content after drying was about 1%. After desiccation, the ampoules were filled with dry nitrogen and sealed. In order to

The Effect of Hydrogen Ion Concentration upon the Preservation of Lyophilised BCG
(*The Figures Represent the Negative* Log_{10} *Titer of Growth*)

Series No.	*pH*	*Immediately after Lyophilisation*		*After 8 Weeks' Storage at 4°C.*		*After 8 Weeks' Storage at 38° C.*	
		3 weeks	*5 weeks*	*3 weeks*	*5 weeks*	*3 weeks*	*5 weeks*
1	4.48	2	3	1	2	No growth	1
2	5.08	2	3	2	2	No growth	1
3	5.59	2	3	2	3	1	1
4	6.11	3	4	2	3	1	2
5	6.50	3	5	3	5	2	2
6	7.05	3	7	2	6	3	5
7	7.42	3	6	3	5	1	1
8	8.12	2	3	2	3	1	1
9	8.56	2	3	2	2	No growth	1
Control*	7.05	5	9				

* Unlyophilised.

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determine the highest dilution at which growth of freshly prepared vaccine suspension and freeze-dried vaccine would still occur duplicate titrations were carried out in Youman's medium. Details and results of the experiments are given as follows : (1) The effect of hydrogen ion concentration on the preservation of the freeze-dried vaccine was investigated using the strain 858 of an 11-day old Sauton culture which was ground in M/15 phosphate buffer at pH7.0 for 25 minutes. The bacterial concentration was the same as above. After centrifuging, the bacterial pellet was twice re-suspended and washed in phosphate buffer solution at a pH at which the drying was to be carried out. Finally the bacilli were re-suspended in 1/5th volume of appropriate buffer solutions. Three microlitres out of four available were used for freeze drying. The table reproduced on the previous page gives the results indicating that there is a decreased growth on either side of neutrality. (2) The effect of the age of culture on the ability of the freeze dried vaccine to withstand storage has been investigated. Result : The older the culture the lower is the ability to withstand storage. (3) The influence of room temperature on the viability of non-freeze-dried vaccine suspensions was investigated. Three suspensions were stored at room temperature shielding them from the direct sunlight. The fourth was stored at low temperature. Result : Only the refrigerated vaccine maintained its titer. (4) The effect of bacterial dispersion on the efficiency of freeze drying was investigated. Result : The poorer the dispersion the poorer was the efficiency. (5) The importance of the drying adjuvant in combating the loss of viable elements during freeze drying was investigated. The table reproduced below gives the results for the 6 most useful adjuvants and for 1 poor adjuvant. An efficiency

Negative Log_{10} in Titer Immediately after Lyophilisation, in the Presence of a Selected Series of Adjuvants

Adjuvant	*Number of Strain*									
	858	425	805	31	873	B59	826	862	186	170V
Galactose d ...	2	2	1	3	2	3	2	4	1	3
Glucose	1	1	1	2	2	1	1	2	1	2
Sucrose	2	1	3	2	2	3	3	2	3	2
Lactose	1	1	1	2	1	2	2	2	1	2
Sodium glutamate	1	2	3	2	1	1	1	2	1	2
Sodium aspartate	2	1	2	3	2	2	4	1	2	3
Gelatin	5	5	4	6	5	7	4	3	5	6

value was allocated to each adjuvant by adding together the log_{10} decrements in titer for the 10 strains of microbacteria. Fixing the desirable efficiency value at 25 or lower only 6 adjuvants out of 62 proved to have the required efficiency, four of these were carbohydrates (galactose, glucose, sucrose, lactose) and two were salts of organic acids, (sodium glutamate, sodium aspartate). (6) The optimum concentration of adjuvant solutions was investigated. Result : The concentration of the carbohydrates made little difference to the outcome of the process except for very low concentrations. In the case of the salts, however, there was a definite optimum range of concentration. (7) The ability of freeze-dried BCG to withstand prolonged storage at low and at high temperatures was investigated. Result : Carbohydrates gave the best protection at low temperature but only 2 adjuvants, the salts mentioned before, gave protection at high (incubator) temperatures. (8) The effect of a combination of adjuvants on storage stability was investigated but no beneficial effects could be observed. The experiments demonstrate generally the difficulty of stabilising BCG without adjuvants but no conclusive information is yet available on the nature of the action of the adjuvant.

Sommaire : On a etudié la stabilité du vaccin BCG lyophilisé et les effets des additifs chimiques.

Article by R. Miller & K. Goodner *Yale J. Biol. Med.* 25, Feb. 1953 262-283

30/IV

Dehydrated Meat

United Kingdom. This report of the Department of Scientific and Industrial Research gives information on vacuum drying and freeze drying of meat in the following chapters : Introduction, Types of Driers and Storage Tests. The research work started in 1938 and it was soon established that the freeze drying of minced raw meat gives satisfactory results but large scale application of the freeze drying process would have required several years of development. In view of the conditions imposed by the war other methods had to be investigated. Extensive experiments with vacuum melter driers normally used for rendering fats and for the production of meat meal from inedibles are reported. This apparatus consists essentially of a steam-jacketed horizontal cylinder fitted with a paddle agitator system. The operational pressure is about 19-25 inch. Hg. Beef and pork were processed in the plant but the results were not better than those obtained by air drying and discontinued. A brief reference is made to vacuum plate drying. The freeze drying equipment used by the Research Station is described in detail. The method employed is shown in the British Patent 539,477. The cylindrical vacuum chamber, 24 inch. high and 14 inch. diameter, was made of glass and mounted vertically on a steel base plate. A condenser held at –35°C was situated in the centre and operated with cold brine. The meat was placed in dishes supported by electrically heated aluminium stages. The operational vacuum was below 1 mm. Hg. During drying the material was kept between –17 and –10°C. The temperature of the material rose as drying progressed but it was not allowed to exceed 20°C. The moisture content of the material was reduced to 2% or

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less. Thick slices of raw beef retained their original volume, form and colour and re-absorbed over 90% of the original water content but showed considerable shrinkage when roasted or grilled. Minced meat showed less shrinkage. Freeze-dried raw minced beef had a sponge like structure. Its density was lower than that of air-dried meat but more consistent. The oxygen uptake by freeze-dried and air-dried meat of varying moisture content in the can was recorded against the oxygen available per gram of meat in the container. The presence of moisture seemed to retard rather than accelerate the development of oxidative taints in the fat. At high moisture contents the non-fatty constituents of the meat appeared to oxidise at a high rate. As a rule canned dehydrated meat was capable of absorbing more oxygen than is available in an airtight container. The effect of moisture content on the keeping qualities of freeze-dried meat are shown in a table reproduced below. At the abnormally high temperature of 37°C the meat of lowest moisture content showed the best keeping properties

Effect of Moisture Content on the Keeping Properties of Freeze-Dried Raw Beef

Moisture Content (%)*	*Oxygen available* (*mg. per g. meat*)	*Flavour Score after Months' at :*							
		37°C. (99°F.)				15°C. (59°F.)			
		1	2	3	5	2	3	5	28
1.5(2.2)	0.41	1.8	2.2	—	—	4.9	3.5	3.6	3.0
1.5(2.2)	0.06	—	5.2	4.3	5.0	5.6	5.7	5.4	5.2
1.5(2.2)	Control	6.0	6.0	5.0	5.5	5.8	5.0	5.5	5.5
4.5(6.7)	0.41	2.8	2.0	—	—	4.3	2.6	3.1	2.3
4.5(6.7)	0.06	—	4.6	2.5	2.5	5.8	4.6	5.5	4.6
4.5(6.7)	Control	5.8	6.0	5.7	5.7	5.8	5.7	5.7	5.4
6.0(9.0)	0.41	3.8	2.8	—	—	3.4	3.5	3.2	2.5
6.0(9.0)	0.06	—	4.4	2.0	2.2	5.5	5.3	5.3	3.7
6.0(9.0)	Control	4.5	6.0	5.5	5.6	5.8	5.5	5.6	5.3

* Figures in parentheses indicate moisture content on the fat-free basis.

retaining its original flavour and texture practically unimpaired whereas the other sample developed a mealy flavour, a slightly drier and crumbled texture. A summary of the conclusions drawn from the experiments is given at the end of the chapter on storage tests. A.o. it is stated that a moisture content as high as 12-15% (on the fat-free basis) may prove to be satisfactory for storage at moderate temperatures.

Sommaire : Rapport du D.S.I.R. sur la lyophilisation de la viande donnant des détails sur les méthodes et les résultats concernant celui-ci et d'autre méthodes de séchage.

Report by Food Investigation Board *D.S.I.R. Special Report No. 57* 1953 2, 22, 26, 41, 98-110

45 — ELECTRONICS — 45

Electrical Breakdown in Vacuum

31/IV

United Kingdom. The experiments described were carried out at a pressure between 1 and 2 × 10^{-5}mm. Hg in a glass vessel of 10 cm. diameter and 18 cm. height. The gap lengths were less than 0.3 mm. Two types of electrode systems were used, a double sphere system and a needle-sphere system. The spheres were 2 cm. in diameter and the needles were fabricated from wires ranging in diameter from 0.015 to 0.062 inch. In a vacuum better than 10^{-3}mm. Hg the value of the breakdown voltage was found to depend on the electrode configuration, on the material of the electrodes and on the state of the electrode surfaces, but not on pressure. The electrodes had been cleaned and polished before use. As repeated breakdowns in a system result in higher voltages to be required for further breakdowns the voltage was allowed to increase in the present experiments until stability was reached, taking the end point as the value of the breakdown voltage to be determined. D.c. and impulse voltages were used. In the former case accurate measurement was difficult. As the voltage increased a pre-breakdown current established itself which grew smoothly as if the gap was acting as a non-linear resistor. There was no visible sign of the onset of breakdown. For the second series of experiments a single stage impulse generator was used giving impulse voltages up to 55 kV with a 1·5/70 microsecond waveshape. A diagram of the circuit is shown. The values of the breakdown voltage measured in this series were reproducible within ±7%. This margin may be due to a change in the state of the surface of the electrodes. In this series accurate values could be obtained without difficulties. The results are shown in diagrams. Invariably, higher values for the breakdown voltage were obtained in the case of the needle-sphere system than in the case of the 2-sphere system for the same spacing. This could be explained by Van de Graaffs theory of breakdown in vacuum. Electrons are liberated from the cathode by ions and photons originated on the anode. The electrons hit the anode and increase the supply of ions and photons, causing breakdown in the end. In the present experimental conditions some photons, ions and possibly electrons may have missed the needle and as a result a higher voltage was required to obtain breakdown. When 0.24 microsecond sweeps were applied to the 2-sphere system fitted with a current-limiting resistor of 10 KΩ at the breakdown voltage of 17 kV, the breakdown occurred at 0.12

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microseconds. In longer sweeps, the current collapsed after it had fallen below 0.25A. When the resistance was changed to 4 KΩ the peak current was 2½ times greater but the extinguishing current was still the same. When the value of the resistor was such that the current could not reach the value of 0.25A no permanent breakdown was produced. The discharge obtained was intermittent.

Sommaire : On a fait des expériences sur les phénomènes d'effondrements électriques sous vide en utilisant un systeme d'électrodes faites de deux sphères et un systeme composé d'une sphère et d'une aiguille.

Paper by D. Leader *Proc. I.E.E.* 100, 11A, No. 3, 1953 138-140

The Electrical Breakdown of Air between Insulators 32/IV

United Kingdom. Using a c.r.t. it is shown that the a.c. breakdown current in a glass vessel with external electrodes, containing air at reduced pressures, consists of a number of separate pulses, the number in a half-cycle increasing with the excess voltage. The current flows in pulses because once a breakdown occurs in the gas, charges collect on the glass walls and produce reverse fields which stop the discharge. These wall-charge fields also determine the phase when the pulses occur. Ionisation by collision of the molecules of the gas and secondary emission of electrons from the inner surfaces of the glass walls are the fundamental processes in the discharge. The breakdown conditions are shown to be similar to a d.c. discharge between metal electrodes. A theory is given explaining the shape of the pulses and their peak currents for different voltages and pressures. (*Science Abstracts*)

Article by W. L. Harries *I.E.E. Paper* No. 1424 1952 6 pp.

O *Sommaire* : On a fait des recherches sur les phénomènes d'effondrement électrique.

A New Electron Multiplication Process 33/IV

Abstract No. : 90/I

Secondary Electron Emission from Barium Oxide 34/IV

United Kingdom. The effects of temperature, pulsed and d.c. operation on the secondary emission of barium oxide have been investigated. Special electrostatically deflected tubes were used in the experiments designed to allow the secondary emission surface to rotate through 90° from its normal operating position in order to facilitate the deposition of barium or barium oxide onto it. This arrangement also ensured that the surface was not contaminated by material evaporating during operation from the primary cathode. The films, 10^{-4} to 10^{-6} cm. thick, were prepared by evaporating the barium onto a nickel or molybdenum support and subsequently oxidising them in oxygen at 10^{-2}mm. Hg and 500°C. or in a radio-frequency discharge. Alternatively, barium oxide was directly evaporated from platinum spirals. Thicker coatings, usually 0.1mm., were prepared by spraying the barium carbonate and converting it to the oxide *in situ* by heating in vacuum. The secondary emission coefficient δ was found to depend on the bombarding energy. At a target potential of 40V δ equalled 1 and reached the maximum value at 600V. Evaporated films gave the highest values when pulsed as distinct from d.c. but their values for both types of operation were higher than those of the sprayed coatings which showed no difference. The performance of the evaporated films was independent of the method of preparation and the nature of the supports. When changing from pulsed to d.c. operation, the corresponding change of their values took place 10^{-5} to 10^{-4} seconds after the beginning of the bombardment. The effect of temperature in the range up to 600°C. at 240V bombardment showed that δ rises slowly to a flat maximum at 500°C. but appears to collapse if higher temperatures are reached. However, this effect is not real and is due to space charges resulting from the onset of thermionic emission. Evaporated films 10^{-6} cm. thick showed a co-

O efficient of 2.8 which decreased to 2.2 for 10^{-4} cm. thick films the same as the value for sprayed coatings. This was attributed to a surface roughening of thicker evaporated films due to crystallisation. During operation, for the first 15-30 minutes of the bombardment, complex short time changes occurred but over long periods of continuous running the value of the coefficient fell as the oxide began to show signs of progressive decomposition signifying reduction to metallic barium.

Article by J. Woods & D. A. Wright *Brit. J. Appl. Phys.* 3, Oct. 1952 323-326

Sommaire : L'émission secondaire d l'oxyde de barium est étudiée comme fonction de l'épaisseur de la couche, sa température, la méthode de préparation et la durée de fonctionnement sous bombardement électronique.

Changes in Secondary and Thermionic Emission from Barium Oxide during Electron Bombardment 35/IV

United Kingdom. This article forms the second part of the paper recorded in Abstract 34/IV. The equipment used, *i.e.* the electrostatic deflexion valve and the experimental techniques are the same as before. The secondary emission coefficient δ was again observed at bombarding energies ranging from 240 to 340V. The short-time effects taking place during the first 15-30 minutes of operation were further investigated. The changes of δ with time for evaporated films of different thicknesses were determined by stopping the deposition process at various stages. In the case of the thinnest films, approximately 10^{-6} cm. thick δ first rose during bombardment and then remained stable over periods of ½-2 hours. Thicker films showed a flat maximum followed by a slow decay, the initial value being a little higher. In the case of films some 10^{-5} cm. thick the initial value was lower and decay set in immediately on bombardment. Still thicker films showed similar decay effects independent of the method of their preparation. All films were also examined for their thermionic emission characteristics at 550°C. The thermionic emission seemed to vary with thickness in an analogous manner and showed an increase with time at a rate proportional to the decrease of secondary emission noted. Oxide coatings run at 30V bombarding voltage, i.e. at δ = 1, did not change their secondary emission characteristics as could be proved subsequently by checks at higher target potentials. Thin films were found to recover

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their emission characteristics if the bombardment was discontinued for 20 minutes but thick films did not. Films of intermediate thickness recovered after re-oxidation treatment. The complete recovery of secondary emission could be effected by heating the surface to 600°C. for 2 minutes. At the same time the rate of decay decreased as the operating temperature increased. It was proved conclusively that these changes in emission were due to the formation of excess barium in the oxide in the region penetrated by the incident beam. The excess barium was destroyed and the secondary emission restored when oxygen was admitted. These conditions were confirmed in experiments with barium evaporated onto the oxide.

Sommaire : Cet article est la suite de l'article mentionné dans le resumé No. 34/IV et traite essentiellement des changements de l'émission primaire et secondaire ayant lieu durant l'opération prolongée en courant continu.

Article by J. Woods and D. A. Wright *Brit. J. Appl. Phys.* 4, Feb. 1953 56-61

New Dispenser Type Thermionic Cathode — 36/IV

United States. A brief report is made of improvements in the design and manufacture of the ' L-Cathode ' the basic principle of which was explained in Abstract 53/I. In the new design, which is known as the ' impregnated cathode ', the cavity below the tungsten surface containing a mixture of alkaline earth carbonates has been eliminated. Instead, a mixture of normal and basic barium aluminates is dispersed within the pores of the tungsten body. Coupled with the application of a new technique for the machining of tungsten cylindrical cathodes of the new type can be manufactured to dimensions as small as 0.06 inch in length, 0.03 inch inner diameter and 0.04 inch. outer diameter. Compared with the previous design more space is available accommodating larger heaters resulting in longer heater life. Furthermore thermal efficiency and homogeneous temperature distribution across the emitting area are improved. Finally the use of the aluminates reduces the evolution of gas from the cathode during processing, reducing the time required for pumping.

Sommaire : Modification de la cathode L facilitant la fabrication de cathodes plus petites et l'usage de filaments de chauffe ayant une durée beaucoup plus longue.

Letter by R. Levi *J. Appl. Phys.* 24, Feb. 1953 233

The Synchrocyclotron at Amsterdam. IV. Details of Construction and Ancillary Equipment — 37/IV

Holland. The synchrochcylotron at Amsterdam has been in continual use for three years. The general scope and a description of the equipment has been given in three previous articles. Here only details of the construction are discussed. The acceleration chamber is 2 metres in diameter. The top and bottom, made of the same material as the magnet, is 7 cm. thick and the sides are made of brass or non-magnetic material. Rubber gaskets were used to obtain airtight joints. In some cases crossed joints were unavoidable and worked well provided one round and one flat rubber was used. The total leakage of the assembly ($2m.^3$) was less than 10^{-5} l.-at./sec. The inner faces of the top and bottom are copper-clad as well as water-cooled. The Dee jackets are also water-cooled. The ion source contains a nozzle, 60 mm. long and 5 mm. in diameter, directed downwards in line with the magnetic field through which gas (heavy hydrogen) enters the acceleration chamber at the rate of 0.3 l.-at./h. The pressure inside the ion source is 0.1 mm. Hg. When −450V is applied to the tungsten cathode a gas discharge develops which causes incandescence of the tungsten and emission of electrons. The intense magnetic field causes the electrons to move in narrow helices giving rise to a highly concentrated electron beam to issue from the nozzle. This passes through the acceleration chamber and causes considerable ionisation even though the gas pressure in the chamber is low. The ion source was built to an empirical design. Its operating mechanism has not been fully clarified yet but a beam current of 20 μA is obtained. The gas flow to the source is controlled by a needle valve fitted to a buffer volume maintained by a bellows type pressure switch at a preset constant value. With a particle energy of 28 MeV at 20 μA the beam dissipates 560 W at the target. The heat developed at the target is carried away by cooling water supplied from a constant-head tank. The beam current is monitored by measuring the temperature rise of the cooling water at the target. To ensure a large beam current the gas pressure in the ion source must be high whereas the pressure in the acceleration chamber must always be below 10^{-4} mm. Hg. The duct connecting the two should not be too narrow. In order to meet the various operational conditions the pumping speed was required to be as high as possible. A specially designed three-stage silicon oil diffusion pump was used backed by a single-stage booster and mechanical pump. A photograph and a diagram of the arrangement are given. A plate valve is fitted at the mouth of the main pump and a series of baffles acting as vapour traps are placed in the pump duct leading to the acceleration chamber. The baffles are soldered to the expansion tube of a Freon refrigerator and are kept at a temperature of −30°C. during operation. The trial pumping speed after assembly to the accelerator was 2,200 l./sec. at 10^{-4}mm. Hg for air and correspondingly higher for hydrogen. Philips gauges were used to measure the pressure and served at the same time as actuators for a variety of control devices. The energising current for the magnet is automatically stabilised with a mirror galvanometer photocell system.

Sommaire : On donne des détails du synchrocyclotron d'Amsterdam.

Article by F. A. Heyn and J. J. Burgerjon *Philips Tech. Rev.* 14, March 1953 263-279

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Acta Cryst.	Acta Crystallographica, Ejnar Munksgaard, Copenhagen, Denmark.
Acta Metallurg.	Acta Metallurgica, 57, East 55th Street, New York 22, N.Y., U.S.A.
A.E.C. Reports	U.S. Atomic Energy Commission Reports,
A.E.C.D. Reports	Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
A.E.R.E. Reports	U.K. Atomic Energy Research Establishment Reports, Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1, England.
Amer. Ceramic Soc. Bull.	The American Ceramic Society Bulletin 2525, North High Street, Columbus 2, Ohio, U.S.A.
Amer. Export Industr.	American Exporter Industrial, Johnston Export Publishing Co., 386, Fourth Avenue, New York 16, N.Y., U.S.A.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 1155, Sixteenth Street, N.W., Washington 6, D.C., U.S.A.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Arch. Tech. Messen.	Archiv für Technisches Messen, R. Oldenbourg Verlag, Lotzbeckstrasse 2b, München, Germany.,
Atomics	Leonard Hill Ltd., Stratford House Eden Street, London, N.W.1, England.
Bild & Ton	Henschel Verlag Kunst und Gesellschaft, Oranienburger Strasse 67, Berlin, N.4, Germany.
Brit. Gel. & Glue R.A.	British Gelatine and Glue Research Association, 2a, Dalmeny Avenue, Holloway, London, N.7, England.
Brit. J.Appl.Phys.	British Journal of Applied Physics, The Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
B.S.I.R.A. Bull.	British Scientific Instrument Research Association, Information Department, 'Sira', Southill, Elmstead Woods, Chislehurst, Kent, England.
Bull. Inst. Int. du Froid	Bulletin de l'Institut International du Froid, Institut International du Froid, 177, Boulevard Malesherbes, Paris, 17e, France.
Canad. Chem. Process.	Canadian Chemical Processing, 137, Wellington Street West, Toronto, Ontario, Canada.
Canad. J. Phys.	Canadian Journal of Physics, The National Research Council, Ottawa, Canada.
Canad. J.Technol.	The Canadian Journal of Technology, The National Research Council, Ottawa, Canada.
Chemical Abstracts	The American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. Engng. Progr.	Chemical Engineering Progress, American Institute of Chemical Engineers, 120, East 41st Street, New York 17, N.Y., U.S.A.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Chemie Verlag, G.m.b.H., Hauptstrasse 129, Weinheim/Bergstrasse, Germany.
Compr. Air Mag.	Compressed Air Magazine, Compressed Air Magazine Co., Phillipsburg, N.Y., U.S.A.
Conference Opt. Soc. Amer.	Conference of the Optical Society of America, c/o Journal of the Optical Society of America, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
C.R. Acad. Sci., Paris	Comptes Rendus des Séances de L'Academie des Sciences, Messrs. Gauthier-Villar, 55, Quai des Grands-Augustine, Paris, France.
C.R. Comm. 1 & 2 I.I.F., Louvain	Comptes Rendus Commissions 1 & 2, Institut International du Froid, c/o Bulletin de l'Institut International du Froid, 177, Boulevard Malesherbes, Paris, 17e, France.
Design & Res. Centre Bull.	Design and Research Centre Bulletin, Design and Research Centre, Goldsmiths' Hall, Foster Lane, London, E.C.2, England.
Drug. & Cosm. Ind.	Drug and Cosmetic Industry, Drug Markets Inc., 101, West 31st Street, New York 1, N.Y., U.S.A.
D.S.I.R. Bibliography (& Reports)	Bibliography of Industrial Drying, Department of Scientific and Industrial Research, Her Majesty's Stationery Office, P.O. Box 569, London, S.E.1, England.
Electronics	McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Endeavour	Imperial Chemical Industries Ltd., 26, Dover Street, London, W.1, England.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.

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Exp. Cell Res.	Experimental Cell Research, Academic Press Inc., 125, East 23rd Street, New York 10, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc. 330, West 42nd Street, New York 36, N.Y., U.S.A.
Giesserei	Giesserei Verlag G.m.b.H, Düsseldorf, Germany.
Glas & Hochvakuum Tech.	Glas und Hochvakuum Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Glastech. Berichte	Glastechnische Berichte, Verlag der Deutschen Glastechnischen Gesellschaft, Untermainkai 12, Frankfurt (Main), Germany.
I.E.E. Paper	Institution of Electrical Engineers Paper (Special Print), c/o Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
Industr. Chem.	Industrial Chemist, St. Margaret's Technical Press, Ltd., 35, Tothill Street, London, S.W.1, England.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Instruments	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
Invest. Phys.	Investigationes Physicae, 40, Carlton Crescent, Summer Hill, Sydney, N.S.W., Australia.
Iron Age	Chilton Co. Inc., Chestnut and 56th Streets, Philadelphia 39, Pa., U.S.A.
Iron & Steel	The Louis Cassier Co., Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Jenaer Jahrbuch	Kommissionsverlag Gustav Fischer, Jena, Germany.
J. Amer. Oil Chem. Soc.	Journal of the American Oil Chemists Society, The American Oil Chemists' Society, 35, E. Wacker Drive, Chicago 1, Ill., U.S.A.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics. 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Chem. Phys.	The Journal of Chemical Physics, American Institute of Physics, Inc., 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Iron Steel Inst.	Journal of the Iron and Steel Institute, Iron and Steel Institute, 4, Grosvenor Gardens, London, S.W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers, Inc. 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J Phys. Radium	Journal de Physique et le Radium, La Société Francaise de Physique, 10, rue Vauquelin, Paris 5e, France.
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards, U.S. Government Printing Office, Washington 25, D.C., U.S.A.
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
J. Sci. Instrum.	Journal Scientific Instruments, Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Lab. Practice	Laboratory Practice, United Trade Press Ltd., 9, Gough Square, Fleet Street, London, E.C.4, England.
Light Metals	Temple Press Ltd., Bowling Green Lane, London, E.C.1, England.
Mater. & Meth.	Materials and Methods, 330, West 42nd Street, New York 18, N.Y., U.S.A.
Mech. World & Engng. Rec.	Mechanical World and Engineering Record, 31, King Street West, Manchester 3, England.
Metal Finish.	Metal Finishing, Finishing Publications, Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Progr.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metal Treatm.	Metal Treatment, Industrial Newspapers Ltd., 49, Wellington Street, Strand, London W.C.2, England.
Metall	Metall Verlag Gm.b.H, Hubertusallee 18, Berlin—Grunewald, Germany.
Metallurgia	The Kennedy Press, Ltd., 31, King Street West, Manchester 3, England.
Mill & Factory	205, East 42nd Street, New York 17, N.Y., U.S.A.

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Mod. Res. Techniques Phys. Metallurgy	Modern Research Techniques in Physical Metallurgy, Report on Conference held in October, 1952, American Society for Metals, Cleveland, Ohio, U.S.A.
Monatsh.	Monatshefte für Mathematik und Physik, Akademie der Wissenschaften, Vienna, Austria.
Mon. Not. Roy. Astr. Soc.	Monthly Notices of the Royal Astronomical Society, Royal Astronomical Society, Burlington House, London, W.1, England.
Nature	Macmillan and Co., St. Martin's Street, London, W.C.2, England.
N.B.S. Report	National Bureau of Standards Report, Office of Scientific Publications, National Bureau of Standards, Washington 25, D.C., U.S.A.
Nederlands Tijdschr. Natuurk.	Nederlands Tijdschrift voor Natuurkunde, Martinus Nijhoff, Lange Voorhout No. 9, The Hague, Holland.
N. J. Cer. Res. Sta. Rep.	Technical Reports, N.J. Ceramic Research Station, Rutgers University, New Brunswick, N.J., U.S.A.
N.R.C. Canada Rep.	National Research Council of Canada Report, National Research Council of Canada, Ottawa, Canada.
Nuclear Science Abstr.	Nuclear Science Abstracts, U.S. Atomic Energy Commission, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
Nucleonics	McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Oak Ridge Nat. Lab. Reports	Oak Ridge National Laboratory Reports, Y-12 Area, Oak Ridge, Tenn., U.S.A.
Optik	Wissenschaftliche Verlagsgesellschaft, m.b.H, Stuttgart, Germany.
Paint, Oil, Colour J.	Paint, Oil and Colour Journal, Scott Greenwood & Son Ltd., 83-86, Farringdon Street, London, E.C.4, England.
Petrol. Ref.	Petroleum Refiner, The Gulf Publishing Co., Box 2608, Houston 1, Texas, U.S.A.
Pharm. Acta Helv.	Pharmaceutica Acta Helvetiae, Schweitzerischer Apotheker-Verein, Sihil Strasse 37, Zürich, Switzerland.
Philips Tech. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, England.
Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Plansee Proc.	Plansee Proceedings, Metallwerk Plansee G.m.b.H, Reutte/Tyrol, Austria.
Plating	The American Electroplaters Society, American Building, 445, Broad Street, Newark 2, N.J., U.S.A.
Proc. Amer. Soc. Test. Mater.	Proceedings of the American Society for Testing Materials, The American Society for Testing Materials, 20th and Northampton Streets, Easton, Pa., U.S.A.
Proc. I.E.E.	Proceedings of the Institution of Electrical Engineers Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road London, S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Royal Society, The Royal Society, Burlington House, Piccadilly. London, W.1, England.
Prod. Finish.	Product Finishing, Sawell Publication Ltd., 4, Ludgate Circus, London, E.C.4 England.
R.C.A. Rev.	R.C.A. Review, R.C.A. Laboratories, Radio Corporation of America, Princeton, N.J., U.S.A.
Rep. Progr. Phys.	Reports on Progress in Physics, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Rev. Mod. Phys.	Review of Modern Physics, American Institute of Physics, Lancaster, Pa., U.S.A.
Rev. Prat. Froid	Revue Pratique du Froid, 254, Rue de Vaugirard, Paris 15e., France.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Science	American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W., Washington 5, D.C., U.S.A.
Science Abstracts	Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
S.P.E. J.	Society of Plastics Engineers Journal, Society of Plastics Engineers, Suite 116-118, 34 East Putnam Avenue, Greenwich, Conn., U.S.A.

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Sprechsaal	Sprechsaal für Keramik Glas Email, Verlag Mueller and Schmidt, Coburg, Germany.
Structural Engr.	The Structural Engineer, Institution of Structural Engineers, 11, Upper Belgrave Street, London, S.W.1, England.
The Times (Industr. Suppl.)	'The Times' Industrial Supplement, 'The Times' Publishing Co. Ltd., Printing House Square, London, E.C.4, England.
Trans. I. Chem. E.	Transactions of the Institution of Chemical Engineers, The Institution of Chemical Engineers, 56, Victoria Street, London, S.W.1, England.
Univ. Calif. Rad. Lab. Rep.	University of California Radiation Laboratory Reports, University of California, Berkeley, California, U.S.A.
Le Vide	Le Vide, Société Française des Ingénieurs Techniciens du Vide, 44, Rue de Rennes, Paris 6e., France.
Weld. J.	Welding Journal, The American Welding Society, 33, West 39th Street, New York 18, N.Y., U.S.A.
Yale J. Biol. Med.	Yale Journal of Biology and Medicine, 333, Cedar Street, New Haven 11, Conn., U.S.A.
Z. Phys.	Zeitschrift für Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Z. V.D. I.	Zeitschrift des Vereines Deutscher Ingenieure, Prinz Georg Strasse 77/79, Düsseldorf, Germany.

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